JOURNAL OF

Metals

TECHNOLOGY · PRACTICE

JULY 1949

Zirconium

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Creep of Copper

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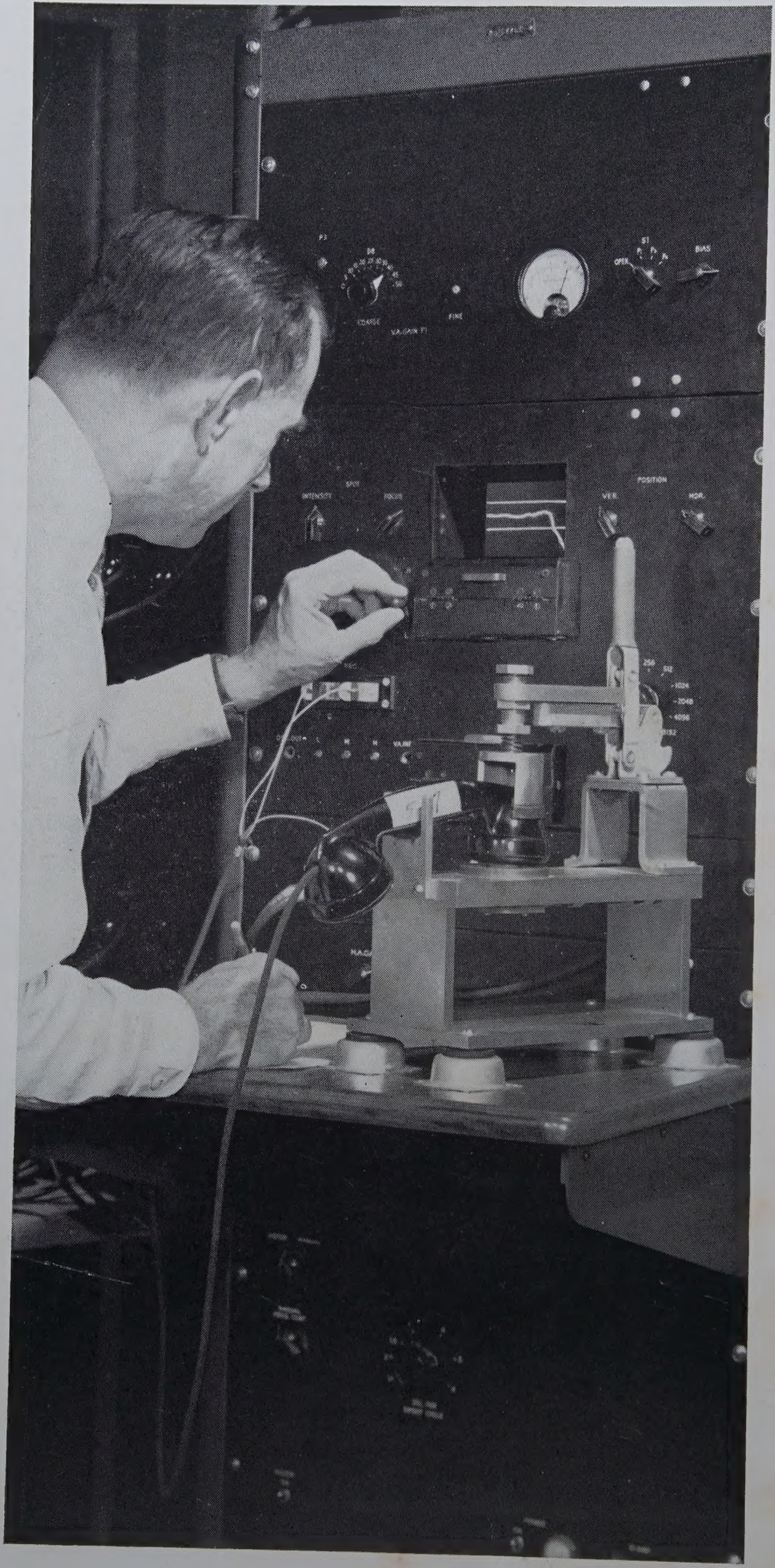
Sintering of Magnetite

A MONTHLY PUBLICATION OF THE
AMERICAN INSTITUTE OF MINING AND METALLURGICAL ENGINEERS
METALS BRANCH



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It listens so YOU can hear better

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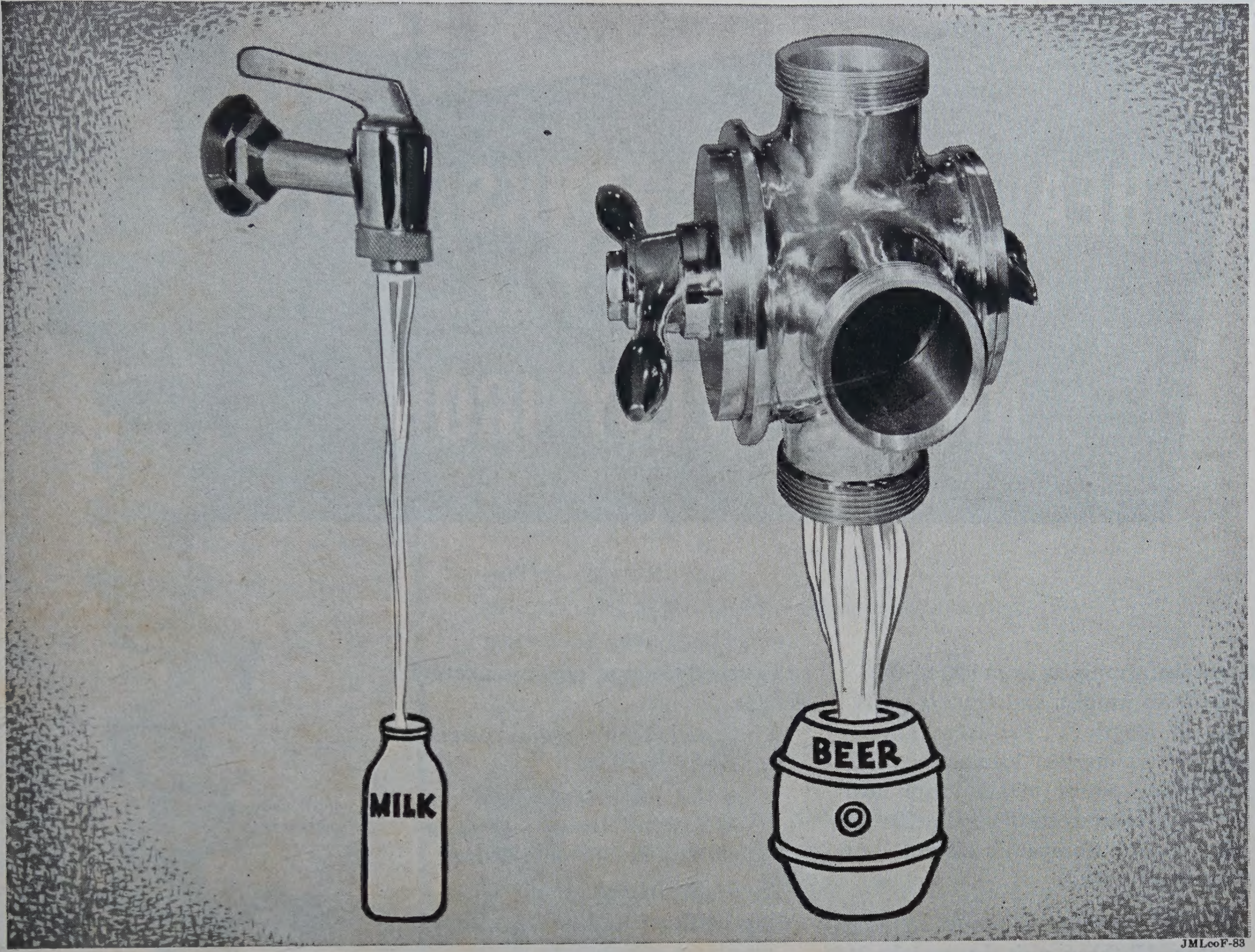

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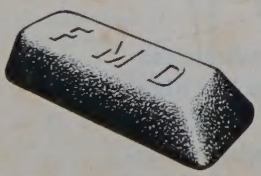

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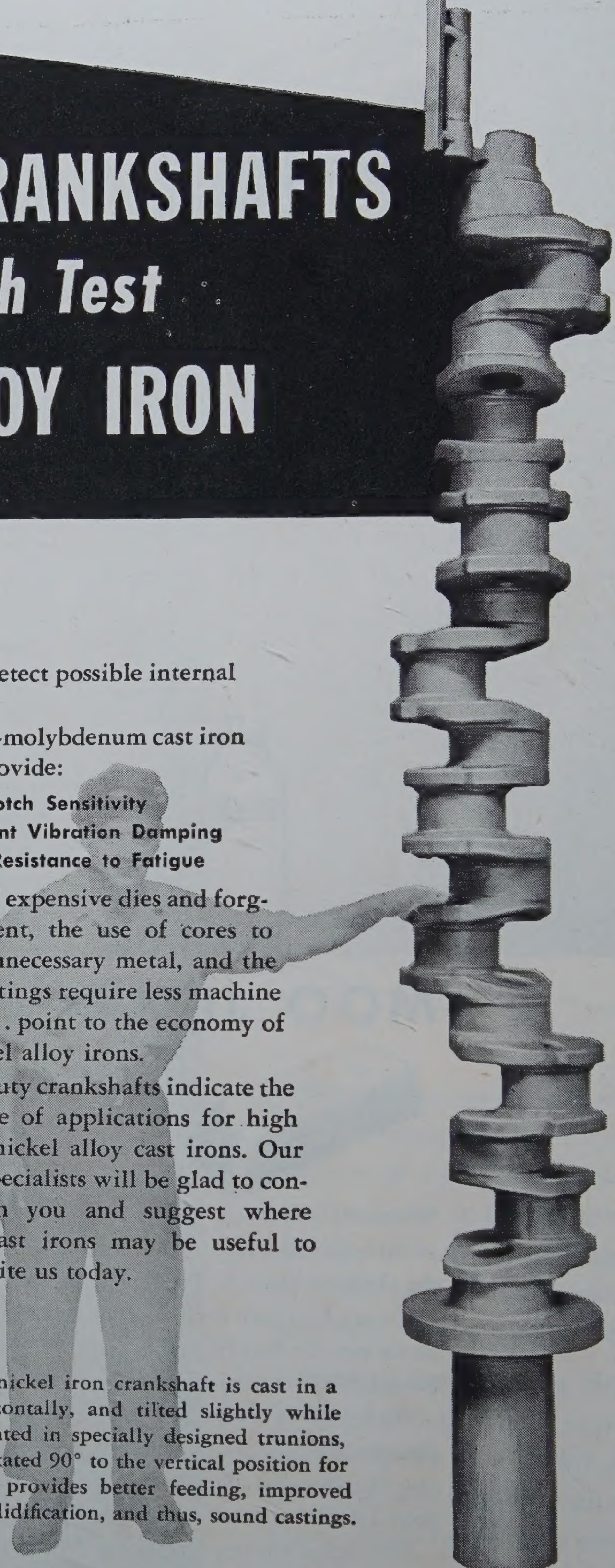

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JOURNAL OF *Metals*

TECHNOLOGY · PRACTICE

July 1949 Vol. I, No. 7

COVER PHOTO: Metallurgy of long ago—Granulating silver or gold (Ercker, 1574). The metals are melted in either the ladle-hearth before the bellows, or in crucibles in the wind furnaces and poured through wet brooms into a tub of rapidly stirred water. The granules are used for subsequent parting by acid, or sulphur, or cementation. A—granulating ladles; B—wind furnaces; D—crucible for melting silver; E—crucible used as ladle; F—copper basin for drying granules; G—iron grate on which to heat silver; H—the granulator, himself; K—the broom man. *(Courtesy of C. S. Smith)*

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AMERICAN INSTITUTE OF MINING AND METALLURGICAL ENGINEERS, INC.
Metals Branch
Editorial, Advertising and Business Office, 29 West 39th St., New York 18, N. Y.
AIME also publishes *Mining Engineering* and *Journal of Petroleum Technology*.

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JOURNAL OF METALS Technology • Practice is on file in many public libraries and is indexed in Engineering Index, Industrial Arts Index, ASM Review and Chemical Abstracts.

Published monthly by the American Institute of Mining and Metallurgical Engineers, Inc., 29 West 39th St., New York 18, N. Y. Domestic subscription, North, South, and Central America, $8: foreign, $9. To AIME members, $6, or $4 in combination with a subscription to *Mining Engineering* or the *Journal of Petroleum Technology*. Single copies, 75 cents. Special issues, $1.50.

Entered as second class matter January 12, 1949, at the post office at New York, N. Y., under the Act of March 3. 1879. Registered cable address, AIME.

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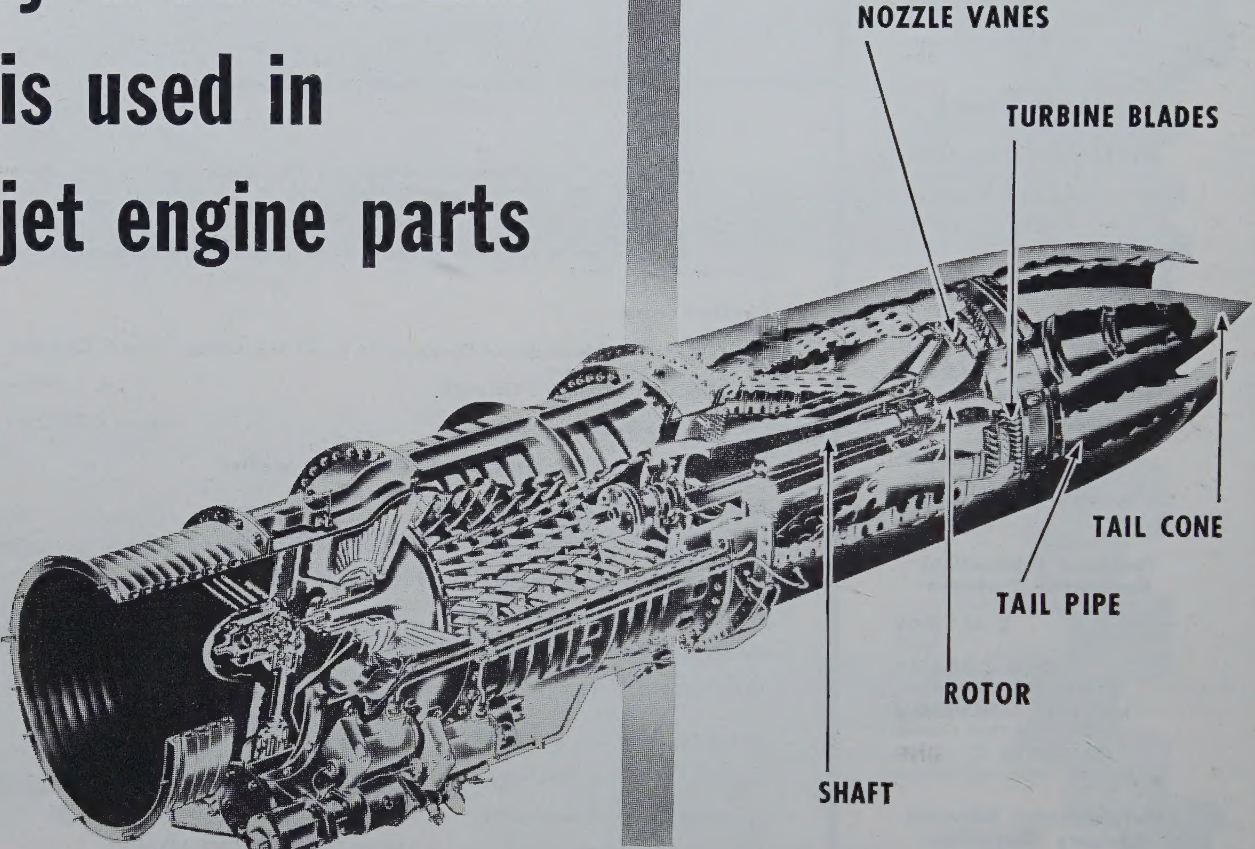
Printed in U. S. A.

why TUNGSTEN is used in jet engine parts



Small amounts of tungsten are used in the alloys for turbo-jet parts because tungsten increases their hardness and strength even at very high temperatures. The percentage of tungsten in high-temperature parts depends upon the physical properties desired and the service temperatures to be met. Tungsten-bearing alloys are produced in both cast and wrought forms.

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GUEST EDITORIAL

KARL T. COMPTON, CHAIRMAN, RESEARCH AND DEVELOPMENT BOARD

THE RESEARCH AND DEVELOPMENT BOARD AND METALS

Coordination of military research and development is the keynote of the Research and Development Board of the National Military Establishment. Its task is accomplished through the combined efforts of a comparatively small working staff composed of the Board Secretariat along with the Committee Secretariats and a larger staff of civilian and military specialists throughout the country who serve on eighteen Board committees. These committees have approximately one hundred panels which furnish them technical advice.

The seven-man Board is composed of two generals each from the Army and the Air Force and two admirals from the Navy, plus myself as chairman. Representing the Army are General Jacob L. Devers, Chief, Army Field Forces, and Maj. General Charles G. Helmick, Deputy Director for Research and Development, Logistics Division, General Staff; Air Force members are General Joseph T. McNarney, Commanding General, Air Materiel Command, Wright-Patterson Air Force Base, and Brig. General D. L. Putt, Director, Research and Development Office, Deputy Chief of Staff for Materiel; Vice Admiral J. D. Price, Deputy Chief of Naval Operations (Air), and Rear Admiral Albert G. Noble, Chief, Bureau of Ordnance, of the Navy complete the membership.

The Board operates in accordance with its directive signed by the Secretary of Defense in 1947 which amplified the basic mission of the Board as it was set forth in the National Security Act of 1947. It succeeds the *Joint* Research and Development Board which was chartered in June 1946 by the Secretaries of the War and Navy Departments to coordinate research and development for the two Departments.

Cooperation by the RDB with other agencies also interested in research and development programs permits economical use of manpower. In the case of the National Advisory Committee for Aeronautics, three members of our Board serve on the 17-member committee. And two members of the NACA, as well as their director of aeronautical research, are members of the Board's Committee on Aeronautics. Intercommunication with the Atomic Energy Commission is achieved by having its Military Liaison Committee, with the addition of four civilian members, constitute itself as the Committee on Atomic Energy on the RDB for consideration of research matters in the atomic energy field. Other agencies with which the Board has continued liaison are the National Security Resources Board, which advises the President concerning the coordination of military, industrial, and civilian mobilization, and the National Research Council of the Academy of Sciences, which carries on research for all Government Departments as requested.

Directors of research in organizations such as the Socony-Vacuum Oil Company, industrial experts such as the president of Thompson Products, Cleveland, executives of the Bell Telephone Laboratories, university presidents, and civilian and military industrial specialists of the National Military Establishment, as well as executives of other Government agencies, are contributing their knowledge and experience to achieve the goal by serving on a part-time basis as chairmen and members of Board committees. At scheduled meetings, they review plans drawn up by the full-time committee secretariats in the Washington headquarters of the Board and determine the program to be followed in accomplishing the work of their respective committees.

These men afford the RDB valuable contacts with scientific societies such as the American Institute of Mining and Metallurgical Engineers, the American Iron and Steel Institute, the American Society for Metals, and the American Society for Testing Materials which enable the Board to be alert to current trends.

Illustrative of the manner in which the Board works is the development of its program in metallurgy under the cognizance of the Panel on Metallurgy. This Panel, with Dr. Clyde Williams, director of Battelle Memorial Institute, as chairman, is under the direction of the Committee on Basic Physical Sciences, headed by Dr. W. V. Houston, president of Rice Institute, Houston, Texas. During the first year the Panel gave much attention to the current metals research and development programs which are aimed at meeting specific military requirements. The metals and their new alloys must be considered from the standpoint of availability, economics, and properties such as resistance to corrosion, wear qualities, weight-strength ratio, formability, resistance to high or low temperatures or sudden changes in temperature, coefficients of expansion, weldability, stresses and mechanical behavior.

Since these new alloys may well be used in aircraft parts, cases, and frameworks for electronic equipment, guided missile components, and small arms weapons or mortar bases they are of interest alike to the Committees on Aeronautics, Electronics, Guided Missiles, Ordnance, and Equipment and Materials, all of which participate in the Metallurgy Panel's program.

Metals for use at high temperature are of particular interest now because of the emphasis being placed on power by propulsion. The objective is to obtain super alloys that will have the same properties at high temperature that carbon steels have at room temperature. The metal requirements will be much the same whether the use is for guided missile propulsion, supersonic bombing propulsion, submarine propulsion, aircraft propulsion, or naval propulsion. For the last 30 years alloys have been used at high temperatures, but the majority of applications have been such that warping and buckling could take place without damage. The new precision propulsion uses require alloys that will not deform when operating temperatures and stresses are applied.

As improvements are made and new uses are found for metals and their alloys as a result of military research and development, industry will be able to adapt them to its needs. For example, propulsion engines now being developed by the military Departments may result in power units that will be of great use in industry. This is some recompense to industry, which carried on a third of the basic research in the country from which the military Departments benefit.

Cooperation among members of industry, universities, the military, and the government agencies engaged in research and development is the heart of the Research and Development Board's task—coordination of military research and development.

(Modern Industry Photo)

Zirconium Forms and Articles Fabricated from Iodide Zirconium Rods, Made by the Foote Mineral Co. An As-deposited Rod is Shown in the Center.

Zirconium Metal

Iodide-, Magnesium-reduce

R. I. JAFFE

There are three kinds of malleable zirconium, which are available commercially or are in developmental stages:

1. Iodide zirconium, which is made by the thermal decomposition of zirconium iodide on a hot filament. It is manufactured by the Foote Mineral Co.
2. Magnesium-reduced zirconium, made by the Kroll process of reacting molten magnesium with zirconium chloride. It is being produced on a rather large-scale pilot plant by the Bureau of Mines.
3. Calcium-reduced zirconium, made by reduction of zirconium oxide with calcium. This process is being developed by the Westinghouse company.

A fourth kind of malleable zirconium is that produced by the Hunter process of reducing zirconium chloride with sodium, a process used by Titanium Alloys Mfg. Co. Very few data are available on the properties of this kind of zirconium, and it will not be discussed in this report.

The cold malleability of zirconium is markedly influenced by the presence of oxygen, nitrogen, and hydrogen in solid solution. Of these impurities, hydrogen is least harmful, because in the production of compact or fused metal in each of the processes there is a vacuum step in which the hydrogen is pumped off. The presence of oxygen and nitrogen is quite different. Once these are present, there is no known method of removing them. Although carbon is picked up by zirconium, it is possible to keep it to a low figure, and, since it does not appear to be appreciably soluble in zirconium, it acts only as a hard ZrC inclusion, and hence has only a moderate embrittling effect. It is of interest to tabulate the oxygen, nitrogen, and carbon contents of the three kinds of zirconium, as shown in Table 1.

TABLE I

Typical Analyses of Zirconium for Oxygen, Nitrogen and Carbon

Kind of Zirconium	Oxygen, %	Nitrogen, %	Carbon, %	References
Iodide Zr (Foote)	0.03	0.01	0.01–0.05	(1)
Mg-reduced Zr (Bureau of Mines)	0.08	0.04	0.15	(2)
Ca-reduced Zr (Westinghouse)	0.13	0.06	Not given	(1)

References:

(1) Lilliendahl, Wroughton, and Gregory: Jour. Electrochemical Society, *93*, June, 1948, 235-246

(2) "O" from above. "N" and "C" from Kroll, Anderson, et. al: Jour. Electrochem. Soc., *94*, July, 1948, pp. 1-20.

It is apparent that iodide zirconium has the lowest oxygen and nitrogen content, Bureau of Mines zirconium next, and Westinghouse the highest; the ductilities are in the same order, iodide best and Westinghouse poorest. Oxygen and nitrogen are deleterious, because they form extensive solid solutions in zirconium, oxygen being reported by Hall, Martin, and Rees (Trans. Faraday Soc. 41, 306, 1945), to be soluble to as high as 50 atom per cent, and nitrogen estimated by van Arkel (Reine Metalle), to be soluble to at least 20 atom per cent.

The tensile properties of iodide zirconium swaged rod are given in Table 2. The tensile strength of the annealed metal is rather modest, and the ductility is not particularly high. The increase in strength on cold swaging 75 per cent reduction in area is considerable (tensile 2½ times as great, yield 4½ times as great), and a significantly large amount of elongation still is left. In connection with the properties given for wire it should be noted particularly that the tensile strength of the swaged 24-gauge wire made by N. V. Philips Co., while checking the strength of the ⅛-in. swaged Foote rod, is considerably lower than the strength of the drawn 25-gauge Foote wire. Raynor did not given any reason for this difference, but one may conjecture that it came about from the method of fabrication used in the wire drawing.

Zirconium has extremely bad seizing characteristics dur-

as of 1949

and Calcium-reduced Zr

BATTELLE MEMORIAL INSTITUTE

Top of Furnace for Production of Sponge Zirconium by Magnesium Reduction of Zirconium Tetrachloride at Bureau of Mines, Northwest Electrodevelopment Laboratory, Albany, Ore. Ingot of Dense Purified Zirconium Chloride is Shown Condensed on Water-cooled Cores Attached to Floating Top.

ing wire drawing, and it has been necessary to resort to expedients like oxidizing (or electroplating) the wire so that the oxide film instead of the metal will bear on the die. It is suggested that the contamination of the wire by dissolved oxygen may have caused the higher strength. The difference in elongation between the Foote value of 26 per cent annealed swaged rod and the Philips value of 11 per cent for annealed swaged wire may be the result of the difference in gage length used, 1 in. or 8 diam. for Foote Mineral Co. and 15 cm. or 300 diam. for N. V. Philips Co.

TABLE 2

Mechanical Properties of Zirconium

Material	Condition	Properties	Reference
Iodide Zr (Foote Mineral or N. V. Philips)	⅛ in. swaged rod annealed at 790 C.	T.S. 36,000 p.s.i. 0.2% Y.S. 16,000 p.s.i. Elong. in 1 in. 26% Red. Area 32% Mod. Elast. 11,400,000 p.s.i. Hardness 30.3 R_B (67 Brinell)*	(1)
	⅛ in. swaged rod 75% reduction	T.S. 88,000 p.s.i. 0.2% Y.S. 70,000 p.s.i. Elong. in 1 in. 9% Red. Area 41% Mod. Elast. 14,500,000 Hardness 87.4 R_B (148 Brinell)*	(1)
	24-ga. swaged wire annealed at 800 C.	T.S. 38,500 p.s.i. Elong. in 15 cm. 11%	(2)
	24-ga. swaged wire reduction not given	T.S. 115,000 p.s.i. Elong. in 15 cm. 3%	(2)
	25-ga. drawn wire annealed at 790 C.	T.S. 61,000 p.s.i. Elong. (gage not given) 14%	(1)
	25-ga. drawn wire, reduction not given	T.S. 129,000 p.s.i. Elong. (gage not given) 2%	(1)
Mg-reduced Zr (Bureau of Mines)	Vacuum melted in graphite crucible	Hardness 68-76 R_B (107-122 Brinell)*	(3)
Ca-reduced Zr	Vacuum sintered	Hardness 188 Vickers (188 Brinell)	(4)
(Westinghouse)	Induction melted	Hardness 280 Vickers (280 Brinell)	

* Taken from Wilson Chart 38, for converting R_B to Brinell (500 Kg lead).

References:

(1) Raynor, Min. and Met. *28*, (1947), 284
(2) Fast, Metalwirt. *17*, (1938), 459
(3) Kroll, Anderson, etc., Jour. Electrochem. Soc., ibid.
(4) Lilliendahl and Rentschler, Tr. Electrochem. Soc., *91*. (1947) 285-297.

The Bureau of Mines has published only hardness data for magnesium-reduced zirconium. However, from its magnitude and the relative ease with which it may be cold worked, one may assume that it is less strong and more ductile than the calcium-reduced zirconium, and stronger and less ductile than iodide zirconium.

No tensile data are available on the calcium-reduced zirconium, as made by Westinghouse. The higher oxygen and nitrogen contents of this metal are reflected in the higher hardnesses shown in the table. Contamination during melting is apparent from the increase in hardness on melting.

Some data are available on the increase in hardness with oxygen content. Lilliendahl and co-workers of Westinghouse using Foote zirconium, and Kroll and co-workers of the Bureau of Mines apparently using their product, have published some figures on this subject which are in good numerical agreement, even though the former used micro-Vickers as a hardness measure and the latter used Brinell-3000 kg. The figures are about the following: 0 per cent oxygen, Brinell 100; 0.1 per cent oxygen, Brinell 200; 0.2 per cent oxygen, Brinell 250; 0.3 per cent oxygen, Brinell 300. Lilliendahl estimates, apparently from experience, that zirconium with over 0.2 per cent oxygen (Brinell 250) is not cold workable. There are no data on the effect of known amounts of nitrogen and hydrogen on the mechanical properties. These elements are known to embrittle zirconium, but specific information is not available.

The ability of zirconium to be cold worked is dependent on its purity. Foote zirconium can be cold rolled, forged, or swaged to reductions over 95 per cent without requiring intermediate annealing. This is remarkable for a hexagonal metal, and one need only realize the limited cold malleability of hexagonal magnesium or berryllium to appreciate it. The rate of work hardening of Foote zirconium has been reported to be high, despite its enormous capacity for cold work. No work-hardening data have been published, however. This impression may come from experience in wire drawing and deep drawing of sheet, where it is necessary to anneal frequently to carry the process along further. The reason for the frequent annealing may be that the rate of work hardening is not high enough, rather than the converse, the material being pulled through the die not becoming strengthened enough by the reduction to overcome the die wall friction and to transmit the force required to deform the metal going through the die.

TABLE 3

Physical Properties of Zirconium

Atomic	(1)	Atomic Number	40
	(2)	Atomic Weight	91.22
	(3)	Lattice Type	H.C.P. Below 880 C. a_o3.22 Å c_o 5.12 Å c/a 1.589
			B.C.C. Above 880 C. a_o 3.61 Å
	(4)	Periodic Classification	Group IV A. 2nd. Long Series
	(5)	Electronic Structure	(2)(8) (18) (10) (2).
Mass		Density at 20 C.:	6.52 g./cc.
Thermal	(1)	Melting point	1860 C. (3380 F.)
	(2)	Linear coefficient of expansion	
		20 – 200 C.	5.4×10^{-6} per °C.
		20 – 400 C.	6.9×10^{-6}
		20 – 700 C.	8.9×10^{-6}
	(3)	Specific heat, 0 – 100 C.	0.068 cal./gm.
	(4)	Thermal conductivity	No data
	(5)	Vapor pressure	No data (B.P. ca. 3000 C.)
Electrical	(1)	Electrical resistivity	39×10^{-6} ohm-cm. (iodide Zr)
	(2)	Electrical conductivity % 1ACS	4.1 (iodide Zr)
	(3)	Temperature coefficient of resistivity	0.004 C^{-1} (iodide Zr)
	(4)	Super conducting at 0.7°K.	
	(5)	Electrochemical equivalent	0.2363 mg./coul.
Thermionic	(1)	Work function	4.1 electron volts
	(2)	Photoelectric threshold, long wave limit	3200 Å
Magnetic	(1)	Diamagnetic, susceptibility	-0.45×10^{-6} at 18 C.

Bureau of Mines zirconium has been markedly improved in recent years in regard to its ability to be cold worked. The metal as originally reported by Kroll and Schlechten in Birmingham, Ala., in 1946, had Brinell hardnesses from 200 to 300, and could only be cold reduced a small amount, perhaps 25 per cent. In the most recent report of the Bureau of Mines given in Columbus in April, 1948, magnesium-reduced zirconium had hardnesses down to 135-140 Brinell, and could be cold rolled at least 60 per cent, and perhaps more. The capacity for cold work was smaller than that possessed by iodide zirconium, however.

Zirconium can be worked in air without danger of oxygen pickup at temperatures up to 600 C., despite the fact that zirconium powder getters oxygen efficiently at 400 C. Adherent oxide films are believed to cause this behavior. At higher temperatures contamination by oxygen and nitrogen will take place, although if the working is done rapidly the contamination will be small.

A successful method of hot working zirconium at elevated temperatures is to first encase the metal in iron or steel cans. After working, the can may be stripped off or dissolved away. The Bureau of Mines reports that 925 C. is the top temperature for this hot-working method. At higher temperatures excessive alloying with iron takes place. The properties of hot-rolled zirconium have not been reported in the literature, but the material probably is slightly stronger and less ductile than vacuum-annealed metal.

Some values of hot tensile strength have been determined by R C A, Harrison, New Jersey; in a helium atmosphere, Foote zirconium wire had the following hot tensile strengths: 500 C—24,000 p.s.i., 700 C.—10,000 p.s.i., 800 C.—6600 p.s.i. This drop in tensile strength with temperature is not particularly severe. Most copper-base alloys lose their strength at as low as 400 C. The retention of strength is not in a class with heat-resisting alloys, however.

In comparing the strength of zirconium with other metals, on an equal volume basis, rolled or swaged iodide zirconium is similar to copper-base alloys. Drawn iodide zirconium and magnesium-reduced zirconium have tensile properties similar to annealed or normalized medium-carbon steel. Since the density of zirconium is 6.5, less than that for these materials, its strength-weight ratio is greater. The strength-weight ratio is not high enough to approach strong aluminum alloys, magnesium, or titanium.

Some of the atomic properties of zirconium are shown in the first part of Table 3. The allotropic change does not have much effect on the heat treatment and subsequent mechanical properties of zirconium, although on passing through it, recrystallization takes place. Quenching or rapid cooling through the transformation temperature produces an acicular microstructure. Zirconium is in Group IV A of the periodic table below titanium and above hafnium. The electronic structure shows that it is a transitional metal with two valency electrons. Another stable configuration for zirconium is (2) (8) (18) (8) (4), with four valency electrons.

The density of zirconium, 6.5, is greater than that of titanium (4.5) above it in Group IV A and less than that of hafnium (13.3) below it. Melting points have been reported for zirconium ranging 1700 C. to 1900 C. The value of 1860 C. reported by the N. V. Philips is generally accepted. No data are available on the vapor pressure of zirconium, but it is believed to be relatively high. The linear coefficient of thermal expansion is rather low, and near enough to certain glasses to have made the Foote Mineral Co. believe some application might be made as a glass-metal sealing metal, but no successful application of zirconium has been made in this field. Also the values for Zr reported from various sources are not in good agreement. With respect to thermal conductivity one would expect it to be low, because of the low electrical conductivity.

The high electrical resistivity of zirconium is about the same as the common resistance alloys, manganin and constantan. Its temperature coefficient of resistance, 0.004 per °C., is a very common figure for pure metals. The values shown in Table 3 are for iodide zirconium; with dissolved impurities the resistivity increases and the temperature coefficient decreases. Thermionic properties exhibited by zirconium are fairly normal for transitional metals of its type.

One of the outstanding properties of zirconium is its excellent corrosion resistance, particularly with respect to HCl, as shown in Table 4. Although not equal to tantalum in its HCl corrosion resistance, it is a close second. In regard to corrosion by bases, zirconium is far superior to tantalum. Except with respect to hydrofluoric acid, aqua regia, hot concentrated H_2SO_4, and hot concentrated H_3PO_4, the acid resistance is excellent. In basic alkaline

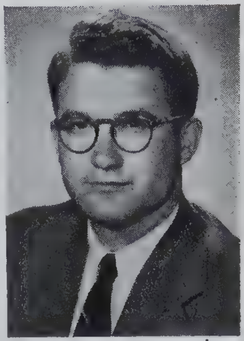
TABLE 4

Corrosion Resistance of Zirconium (Data from Foote Mineral Co.)

	Agent	Concentration	Room Temperature —Inches per year—	100 C.
	Atmosphere	—	No Attack	No Attack
Acids	HCl	5%	No Attack	No Attack
	HCl	Conc.	0.0001	0.0002
	H_2SO_4	10%	0.0002	0.0007
	H_2SO_4	Conc.	—	Attacked
	HNO_3	10%	0.00001	0.00003
	HNO_3	Conc.	0.00001	0.00005
	Aqua Regia	—	Attacked Slowly	Attacked
	HF	—	Rapid Attack	—
	H_3PO_4	10%	0.00001	0.00005
	H_3PO_4	Conc.	0.00004	Attacked
Bases	NaOH	10%	No Attack	0.00002
	NaOH	50%	—	0.00017
	NaOH	Fused	No Attack	
	KOH	10%	0.00002	No Attack
	KOH	Fused	Slight Attack	
Others	KNO_3	Fused	Attacked	
	Cl_2 water		Attacked (embrittled)	
	$FeCl_3$ solutions		Attacked (embrittled)	
	NaCl	20%	—	Slight Tarnish

solutions, there is very little attack. Fused NaOH is without effect, and fused KOH shows slight attack. Zirconium is attacked by fused KNO_3, Cl_2 water, and $FeCl_3$ solutions.

This article was intended to cover only the properties of unalloyed zirconium, with no mention of zirconium-base alloys. It should be pointed out, however, that the field of zirconium-base alloys potentially is a very fruitful one, and many laboratories in this country are working on the problem. For example, magnesium-reduced zirconium, as produced by the Bureau of Mines, is in reality an alloy of zirconium with oxygen and nitrogen in solid solution and having carbides dispersed throughout the grains. These alloying constituents, although present in very small amounts (see Table 1), exert a profound influence on mechanical properties, particularly with respect to strength.



Robert I. Jaffee is a metallurgist in non-ferrous physical metallurgy at Battelle Memorial Institute, where he has been since 1943. He also has been associated with the U. S. Bureau of Mines, Leeds and Northrup Co., and the University of California. He attended Armour Institute of Technology, Harvard University, and the University of Maryland. He is a member of AIME, American Society for Metals, Sigma Xi, Tau Beta Pi, and Phi Lambda Upsilon.

Dr. Jaffee has done considerable experimental work with the lesser known metals, of which zirconium is one. Many of his publications have appeared in AIME Transactions, Institute of Metals. He has written papers on titanium, tungsten, and germanium alloys as well as on alloy studies of the more common metals.

Tin Research Institute Has Been Formed at Columbus

John Ireland, director, Tin Research Institute, London, England, announces the formation of Tin Research Institute, Inc., an American corporation devoted to providing free technical service to consumers of tin in the United States. The office of the new corporation, at 492 West Sixth Ave., Columbus 1, Ohio, will in the future, handle all inquiries or requests for technical service, while a sponsorship will be maintained at Battelle Memorial Institute to inaugurate all new researches.

R. J. Nekervis

R. M. MacIntosh

Technical experts are available for consultation and practical assistance either at the Tin Research Institute or at the consumers' plants. A wide range of publications covering every important use of tin is available free of all charges and inquiries are invited.

Robert J. Nekervis has been appointed supervisor of metallurgical development and Robert M. MacIntosh has been appointed supervisor of chemical development in the new corporation. Both have been associated with Battelle for eight years, and served as assistant supervisors in the nonferrous division handling problems relating to tin.

The trustees of the new organization are Clarence D. Laylin, Dean Charles E. MacQuigg, and Dr. Bruce W. Gonser, all of Columbus.

Eight Alloying Elements Comprise New "Octanium"

The fairly recent alloy developed by the Elgin Watch Co., called "Elgiloy," used for watch mainsprings, is proving quite versatile. It has bobbed up again under a new name, Octanium, as used by the Parker Pen Co., Janesville, Wis., which company will use it for nibs of a new fountain pen which the company is manufacturing. Among other uses for this alloy are springs for rifles, corset stays, and other types of springs. One of the best features is corrosion-resistance.

It also has plenty of strength and long life. Parker Pen Co. has given the alloy a new name for their purposes since their application is supposedly one in the jewelry field which should be distinguished by its own trade name.

The analysis of the alloy is as follows: Chromium, 20 per cent; cobalt, 40; nickel, 15.5; iron, 15; molybdenum, 7; manganese, 2; carbon, 0.15; beryllium, 0.03. Hence, "Octanium" derives its name from the eight alloying elements that comprise it.

Whether our low-grade deposits can be mined to supplant diminishing high-grade reserves will be decided by progress in . . .

. . . Iron Ore Beneficiation

By **FRANCIS X. TARTARON** • MANAGER OF ORE RESEARCH, JONES & LAUGHLIN STEEL CORP., MEMBER, AIME

Beneficiation is the use of technological methods for the improvement of quality and structure of iron ores so as to render them usable in current smelting operations or to improve their usability. In considering the utilization of beneficiation on low-grade iron ores, it is first necessary to examine the need for such treatment. This need is directly related to the reserves of high-grade ore and the possibility of exhaustion of these reserves in the near future. Viewing the iron ore industry as a whole, exact knowledge as to the duration of high-grade reserves and the final technical and economic outcome of exhaustion of these reserves is impossible at the present time. The situation is a complex one involving discovery and location of new ore deposits, the separate activities of large industrial organizations, the national welfare, and finally, the general economic situation in the future. No one knows just what pattern will be finally formed by these factors. But, there is the steady pressure to produce 50 million or more tons of high-grade ore for furnace consumption. Currently about 100 million tons are being produced. If this quantity must be obtained by beneficiation eventually, considerable time is necessary to make adjustments in mining, to determine the most economical flowsheets, to operate pilot plants, and, last but not least, to obtain the necessary finances. Plants must be erected capable of treating 50,000 to 100,000 tons a day with tailing disposal equal to about two thirds of this amount. Quantity of ore mined must be tripled, as compared with present mining operations on the Mesabi Range. The transition is one of stupendous proportions and preparations must be made far in advance of actual need.

Latest figures reveal that the Mesabi Range yields about 65 per cent of the total ore production in the United States. The future of high-grade reserves on the Mesabi Range is necessarily a preponderant factor in the steel industry and in the need for beneficiation. Estimates of these reserves differ sharply but, for purposes of planning, the consensus is that twenty years' duration is a safe limit. The Mesabi Range also furnishes vast reserves of low-grade ore. The so-called "taconite" deposit is estimated to contain some 60 billion tons of crude from which 20 billion tons of high-grade may be derived. The iron mineral, however, is distributed in the ore in a fine particle size and to liberate this mineral prior to concentration, grinding through 100 mesh at least is required. Concentration of this fine material is by the relatively expensive flotation or reduction-magnetic separation process. Thus the utilization of the Mesabi low-grade ores involves a large increase in operating cost, placing them beyond the range of current ore prices. On the eastern end of the Range, however, there exists a deposit of magnetite ore stated to comprise some 5 billion tons from which approximately 1.7 billion tons of concentrate may eventually be obtained. The cheap magnetic concentration process can be employed on this ore. This, together with a sharp reduction in Minnesota taxes on taconite, has apparently brought the magnetic taconite ore within the realm of present economic conditions. At the present time, a proving plant, operated by the Pickands-Mather Co., is in operation at Aurora, Minn., treating 75 tons per hour of magnetic taconite. Grinding is through 100 mesh. Twenty-five tons per hour of concentrate are produced and pelletized by the Minnesota pelletization process.

Thus, by focusing on the Mesabi Range, we have a core of knowledge concerning the future of the iron ore industry. There are, roughly, twenty years to prepare for exhaustion of the high-grade ores. A vast quantity of low-grade ore exists which on beneficiation can produce the tonnage required for the furnaces. On the eastern end of the Range, a portion of this ore is of such nature that it may be treated under present economic conditions. Hence, the transition from high-grade to low-grade is already taking place.

Michigan's low-grade ore is also the taconite type, hematite being the predominant iron mineral. Reserves have recently been estimated to be about 2 billion tons from which some 725 millon tons of concentrate may be derived.

The preceding considerations were devoted to the broad picture of iron ore production, the objective being the maintenance of flow of 50 million or more tons of ore to the furnaces. When, however, we turn to the problems of the individual industrial organizations, a much more urgent situation is revealed. It is not a matter of twenty years but of one or two years and methods of beneficiation employed must be clearly economic and immediately applicable. The problem in these cases is not the total exhaustion of reserves, but the maintenance of production schedules. As one deposit is exhausted, another must take its place. The type ore subjected to beneficiation in such cases is known as "intermediate-type" ore. The salient characteristic of such ore is that fine grinding is not required; concentration is performed on relatively coarse particle mixtures, hence, cheap treatments can be employed. In the Minnesota and Alabama ore fields, gravity concentration predominates, whereas in New York and Pennsylvania, magnetic separation methods are employed. Negligible concentration is practiced in Michigan at the present time.

In terms of average production of iron ore in the United States from 1942 to 1946 about 15 per cent represents beneficiated intermediate ore from the Lake Superior district, three per cent from the Northeastern district, and one per cent from the Southeastern district. Thus the bulk of the concentrate comes from Minnesota. Reserves of Minnesota crude intermediate-type ore are estimated at 300 million tons of producible concentrate, those of the Northeastern district at 500 million tons of crude. In the overall picture of the iron ore industry, the intermediate-type ores are not important due to relatively low reserves available. But, they are supplying current needs at current prices and at present are filling the gap between supply and demand.

nature of beneficiation processes

The raw materials of beneficiation are, in the main, the products of nature occurring at or near the surface of the earth. While there is considerable variation in chemical composition and physical form of these products, yet from the point of view of beneficiation they are treated in accordance with a few simple principles. A low-grade ore is an aggregate of different species or kinds of minerals, the minerals occurring in particle or grain form of varying size. In most cases, these mineral particles are consolidated in a rock which may be original or partly or completely decomposed. In other cases, they may be a simple mixture of free particles of the constituent minerals. Where the particles are consolidated so that there is attachment of particles of different kinds of minerals to one another, severance of liberation is required prior to concentration. This is ordinarily accomplished by crushing or grinding. The size range resulting from this crushing or grinding determines the concentration method as well as the economics of the treatment. For example, at the present time any iron ore requiring crushing and grinding through 325 mesh to effect liberation is questionable as to economic beneficiation under present conditions, regardless of the method of concentration employed.

Concentration is the separation of the particles of one mineral species from the others and this is effected substantially in all methods by means of differential motion. We are dealing with a mixture of particles of different mineral kinds and by imparting motion to one kind different from the others, separation is effected. This is the almost universal method employed in concentration—differential motion of particles of one species of mineral from those of another. This differential motion is usually accomplished by three broad methods: (1) gravity concentration, wherein particles of different specific gravities move differently in a resistant medium such as water or air; (2) magnetic separation, wherein magnetizable mineral particles take different paths in a magnetic field as compared with non-magnetizable mineral particles; and (3) flotation, wherein particles of one mineral kind are reagent coated so that they adhere to air bubbles, and the buoyancy of the bubble-mineral aggregate causes motion to the surface of the liquid whereas the nonadherent particles remain in suspension.

Subsequent to separation, we have agglomeration or reconsolidation of the usable mineral so as to prepare it for the smelting operation. The blast furnace cannot retain material less than approximately ¼-in. size, and where concentrates are too fine, it is necessary to join the particles into larger aggregates. In the size range of ¼ in. to 100 mesh this is accomplished by sintering whereby a moistened mixture of coal and ore is burned by means of air being sucked through the heated mass. Consolidation takes place, forming chunks. Where the ore is preponderantly below 100 mesh in size, the porosity of the bed is too low to permit adequate passage of air. A different method is now being tested on a plant scale. This is the so-called Minnesota pelletization method, whereby moistened concentrate containing fifty per cent or more of minus 325 mesh fraction is rolled into balls by means of a rotating cylinder. These balls are then baked in a shaft furnace which causes hardening and resistance to impact and abrasion. The method is rated as cheaper than sintering and production runs are in progress at Aurora, Minn.

gravity concentration processes

Gravity processes are utilized in Minnesota and Alabama and to a small extent in the Northeastern district. In such processes, the phenomenon that is utilized is that minerals of higher specific gravity settle faster in water than those of lower specific gravity. Various mechanical devices are employed that fit best the characteristics of the ore to be treated. The most important of these characteristics are (1) magnitude of difference in specific gravity of the minerals, (2) size range present after liberation, and (3) shape of the particles.

In a vacuum, all mineral particles regardless of specific gravity, size or shape, move toward the earth with equal acceleration, and if started at the same instant, with equal velocity. Hence, no separation is possible. When, however, such particles are immersed in a resistant medium such as water, differences in settling velocity are distinct. Mineral particles of higher specific gravity settle faster than those of lower specific gravity. This effect, therefore, must be produced by the medium. In a vacuum, the only force operating is the force of gravity, but in a resistant medium, two new forces are introduced, buoyancy and friction. If this situation is analyzed in detail, it will be found that buoyancy is the force responsible for the difference in settling rates between particles of different specific gravities. Furthermore, this unequal settling takes place without regard to size or shape of particle. It is based solely on difference of specific gravity.

Friction, on the other hand, is a force that interferes with the effect produced by buoyancy. It acts against the differential derived from this force. Size and shape of particle are intimately linked to the force of friction and it is because of this that gravity concentration is inefficient and complex. As a result of friction, fine particles of high specific gravity settle equally with coarser particles of lower specific gravity and separation cannot be made. This is known as size interference. In one case this effect of friction is helpful. Where the ore impurity consists preponderantly of fine low-specific gravity mineral, such mineral is slowest settling and easily sepated. In Minnesota, over eighty per cent of the beneficiation is done by simple water washing of the ore so as to remove fine silica and produce marketable concentrate.

The force of buoyancy can be magnified by raising the specific gravity of the medium. This can be done by increasing the proportion of ore solids to water or by adding a separate mineral of very high specific gravity to the ore pulp.

We have then, two situations in gravity concentration: (1) when a mechanical arrangement is so contrived that buoyancy is a predominant factor, then sharpest separations take place and there is negligible size interference; (2) when buoyancy is less predominant, friction causes size interference as well as slowing up of the differential motion as a whole. Separation is much more difficult. In general, friction can be minimized and buoyancy utilized in the coarsest fractions of the feed. It is in the finer portions of the ore that friction predominates and causes interference. To eliminate this interference, it is necessary to divide the feed into narrow size bands within which fine material of higher specific gravity settles faster than the coarser gangue minerals. The finer the ore, the slower the over-all settling rate and the longer the time required to obtain separation. Hence, on finer ores, the capacity of machines decreases and the size and quantity of machines is larger to treat a required tonnage.

In practice, we find that buoyancy is applied on a size range of 1¼ to 3/16 in. This is utilized in the so-called heavy-density process which will be described later. Below 3/16 in., friction enters as so important a factor that only imperfect application of buoyancy is possible. Methods devised to treat the minus 3/16 in. range are full of complications. No single machine can treat the full range; most give only a partial answer. Some give a good answer but are difficult to use. For example, we are finding that the shaking table can concentrate ore from 3/16 in. to 20 mesh and from 20 mesh to 150 mesh when treated separately. Yet this machine is of such small capacity and requires so much floor space to treat a high tonnage, that its application is problematical. The hydrosizer makes a partial concentration on minus 10-mesh feed. The Humphreys spirals handle ore from 10 or 20 mesh to 150 mesh. A new machine claims to concentrate from 3/16 in. to 65 mesh. Another is being tested on 65 to 325 mesh. Below 325 mesh no one has or expects an answer by means of gravity concentration.

The reason for utilizing gravity concentration is that it is cheap. Magnetic separation would be far superior if the iron mineral were magnetite. But with hematite, the only economic method is by gravity processes. Flotation cannot be employed on material coarser than 65 mesh and even on the minus 65-mesh material it is still too expensive.

washing

In Minnesota, there are two types of intermediate ores, one known as wash ore, the other as "jig" ore. In wash ore, the silica particles are much finer in size than the iron mineral particles, hence, a sizing operation can effect concentration. This is simply and cheaply done by suspending the ore in water in a container allowing the coarse material to settle and overflowing the fines. The machines employed are log washers and mechanical classifiers.

Over eighty per cent of Minnesota shipments of beneficiated ore represent wash ore. The remainder is known as "jig" ore, and contains silica particles of the same size range as iron mineral particles. Hence, more elaborate concentration methods are necessary. The name "jig" ore was derived from the machine that originally was employed to treat such ores. The jig is now almost obsolete on the Mesabi Range, having been replaced by the heavy-density process.

heavy-density process

The heavy-density process utilizes the phenomenon that when finely ground mineral of high specific gravity is suspended homogeneously in water, a new liquid medium is formed having a higher specific gravity, higher viscosity, and higher resistance to movement of particles. By varying the proportion of suspended particles to the water, the specific gravity can be raised or lowered. When minerals of different specific gravities are introduced into this medium, the lighter can be made to float and the heavier to sink. However, as the introduced minerals are decreased in size, more and more interference with the separation takes place due to the high resistance to motion and an increase in viscosity brought on by the presence of the fine minerals to be separated. As a result, in plant operations the lower size limit is around 4 mesh or 3/16 in. The upper size limit on iron ores is 1½ in. Within this size range, a highly successful separation is obtained, and the use of heavy density is rapidly expanding on the Mesabi Range.

The material suspended in water is ferrosilicon, having a specific gravity close to 7 and ground through 65 to 100 mesh. Sometimes, magnetite is employed in conjunction with ferrosilicon where low specific gravities are desired. Specific gravity of the liquid medium varies from 2.6 to 3.2.

Apparatus employed consists of two units: the separatory unit and the medium purification unit. The separatory unit consists of screens to size the feed, a cone or a mechanical classifier into which sized ore and medium are introduced, followed by screens to separate the medium from the concentrate and tailing. This medium goes to the purification unit which consists of magnetic separators and dewatering machines. The so-called "densified" medium is then reintroduced into the circuit.

As an example of heavy density separation, the test results in the accompanying table were obtained on a Minnesota ore in our laboratory. It is expected that the same results can be obtained in plant operation.

Results of Heavy-Density Separation Tests on a Minnesota Ore

		Concentrate, Percent		
Specific Gravity of Medium	Percent Weight of Feed	Iron	Silica	Iron Recovery
2.70	90.0	58.48	12.02	96.28
2.85	79.5	59.56	10.14	87.01
3.00	69.4	60.72	8.54	77.02

It can be seen from these results that by adjustment of the specific gravity of the medium, concentrates of different grades can be obtained with corresponding changes in recovery.

magnetic separation

Magnetic separation is employed preponderantly in the northeastern section of the United States including New York, New Jersey, and Pennsylvania. It is based on the phenomenon that the iron mineral magnetite is easily magnetizable, hence, when particles of magnetite are passed through a magnetic field, they take a different path from particles which are nonmagnetizable. The explanation of magnetization is that the molecules or atoms of substances are unit magnets. In magnetizable materials, the molecules can rotate easily, hence, when placed in a magnetic field, north and south poles line up in the same direction. The over-all magnetic field is then the sum of the unit magnetic fields. In nonmagnetizable substances, the molecules cannot rotate, the unit magnets neutralize one another and the over-all field remains at zero or low magnetic intensity. When a substance is magnetized, the space surrounding it becomes energized or activated, and when brought into contact with a similarly activated space, reactions occur which are reflected in the motions of the material bodies to which these activated spaces are linked. Thus, a magnetized bar of steel can be suspended in mid air with no material support. Below this piece of steel is another bar of steel, also magnetized. The space between them is energized and keeps the upper bar suspended in mid air.

Magnetic separation is the cheapest and most efficient of all beneficiation processes. There is no size interference as in gravity concentration except where the feed is composed predominantly of slimes. Here, there is the lumping of the fines with most of the impurities trapped in the lumps. But, most ores do not contain such a predominance of slimes and no serious problem is present. The greatest difficulty with magnetic separation is that the major tonnage of iron ore reserves contains the nonmagnetizable hematite rather than magnetizable magnetite. Thus, the process has limited application. Hematite can be reduced to magnetite by heating and using reducing agents such as carbon monoxide or hydrogen, but the cost of this method is high at the present time. Efforts are constantly being made to reduce this cost to within economic limits.

In the practice of magnetic separation on a high tonnage basis, it has been found that the magnetic field cannot be adjusted so as to make a sharp distinction between free iron mineral and iron mineral attached to gangue. A particle of quartz with ten per cent of its weight as attached magnetite will go into the concentrate together with free iron mineral. Thus, segregation of attached mineral particles into a separate middling product, which is common in other processes, does not take place in the magnetic process. However, advantage is taken of this factor to reduce crushing and grinding cost. The ore is crushed to produce free gangue mineral, considerable locked mineral, and some free iron mineral. It is then subjected to magnetic separation which segregates the free gangue mineral only, which is rejected. The reduced quantity of concentrates is then crushed finer and the process repeated. By this means, less and less quantity of material is subjected to crushing and grinding to the point where complete liberation takes place and final grade of concentrate is attained. The standard flowsheet for magnetic separation is stepwise liberation and concentration based on the characteristic behavior of the magnetic field.

On the eastern end of the Mesabi Range, there is a deposit of magnetite ore running about 25 per cent iron, estimated to contain about 5 billion tons from which 1.7 billion tons of concentrate may be derived. This deposit originally contained hematite, but an intrusion of igneous rock reduced the hematite to magnetite and at the same time consolidated the ore into an extremely hard rock. Drilling for mining operations is expensive and for a long time this deposit was uneconomic to treat. Changes produced by the war and a lowering of state taxes has apparently made it possible to exploit the deposit. Pickands-Mather Co. is now operating a 75-ton-per-hour pilot plant producing high-grade pellets as final product for blast furnace use. If successful, full scale plants will be erected.

Machines currently employed for magnetic separation are of the belt type and the drum type. The belt-type machine has an endless belt, the lower branch of which runs through ore pulp flowing through a compartmented box. Above this portion of the belt is an electromagnet with the north and south poles alternating. As the belt passes beneath the magnet and through the pulp, magnetite adheres to the belt until it passes beyond the magnets. It then drops into the concentrate compartment of the box. Alternating north and south magnets cause rotation of the magnetic particles so as to free trapped gangue.

The drum-type machine has rotating cylindrical drums immersed part way in the pulp with stationary magnets inside the drums. The pulp also flows through a box usually containing three compartments, each with its individual drum, the three drums operating in series. Concentrate from the first drum is retreated by the second, and the second concentrate by the third drum. The drum-type machine appears to be superior to the belt type on fine material and is being used on the magnetic-taconite at Aurora, Minn.

flotation process

Flotation has not yet found application in iron ore concentration due to the fact that it is a relatively expensive process and requires a large investment in plant. However, if taconites are to be beneficiated, flotation will loom as an important method of the future. Hence, it is reviewed here.

In flotation, a suspension in water of ore particles 35 mesh and finer is agitated in a cell through which air bubbles are rising. By adding the proper chemicals to the pulp, mineral particles of one type adhere to the air bubbles and rise to the surface, forming a froth which is skimmed. Thus, one mineral is removed and the other or others remain in the pulp, flowing through a series of cells to the discharge end of the machine.

The adherence of mineral particles to air bubbles is effected by the use of a special type of organic compound having a characteristic chemical composition and physical properties. Molecules of such compounds consist of two primary ingredients. One is a hydrocarbon portion characterized by water repellency and water insolubility as well as chemical inertness. The other is a water-soluble, water-attracted, chemically-active group attached to the hydrocarbon portion. Oleic acid is an example of a common type flotation collector. This compound contains a straight chain hydrocarbon group of seventeen carbon atoms and to it is attached a carboxyl group. At an interface between water and air, such molecules become oriented with the hydrocarbon projecting into the air and the carboxyl remaining in the water. When oleic acid is dissolved in water (it is slightly soluble), the surface of the water can be shown to have the properties of an oil rather than of water. The hydrocarbon portions of the oleic acid molecules concentrate in the surface layer and thus cause the change in properties.

When particles of an ore pulp and flotation collector are mixed together in water, the collector reacts selectively with the mineral particles of one kind and the effect of this reaction is to cause adherence of collector to mineral. Adherence and selectivity are based on chemical phenomena and the whole field of chemistry is available for utilization in devising separations of minerals from one another.

Once a flotation collector has been properly attached to a mineral particle, adherence to air bubbles follows automatically. On contact of bubble and coated mineral particle, the force of water repellency is brought into play and acts to resist separation because separation means re-entry of the hydrocarbon into the water phase.

Frequently a mineral does not react with a collector or if it does, it is desired to suppress this reaction. By the use of auxiliary reagents, it is possible to alter the chemical nature of mineral surfaces so that reactions different from normal are obtained. Thus, iron minerals react with oleic acid-type collectors and normally are floated. By the addition of soluble starch, the iron mineral becomes coated with this material and its reaction with oleic acid prevented. Addition of lime causes alteration of quartz surface so that a reaction takes place with the collector. Quartz now is coated with collector and it adheres to air bubbles. In this way new separations are devised and methods of separation can be developed on any combination of minerals.

Flotation revolutionized the nonferrous metal mining industries because substantially all ores that were liberated at sizes adequate for gravity concentration were depleted years ago. Ores now treated resemble the taconite ores and flotation is the only method applicable. However, on iron ores, the possibility of reducing hematite to magnetite must be considered and it remains to be seen which method will eventually be adopted.

Flotation, when compared with gravity concentration and magnetic separation, is a relatively expensive process. The existence of ore reserves that can be concentrated by these two methods keeps flotation in the background. In the course of time, however, as the high-grade and intermediate ores become more and more difficult to obtain, flotation will emerge as an important method and may become the predominant process.

future of iron ore beneficiation

As indicated in the opening paragraphs, the maintenance of a flow of 50 million or more tons of high-grade ore to the furnaces will involve a stupendous transformation in the iron mining industry when such ore must be obtained by beneficiation. The magnitude of beneficiation operations will increase accordingly. The production of 50 million tons or more a year of high-grade concentrate means the mining and treatment of about half a million tons of crude per 24 hour day. Mining must be performed in hard rock as contrasted with the loose material now being dug. Crushing and grinding must be done so as to reduce boulders 4 to 6 ft. in size down to 100 mesh or finer. Beneficiation must be followed by pelletization. Large scale plants will be necessitated to gain greatest economy and minimize the inevitable increase in cost.

Plant investment to treat taconite-type ores has been estimated at $10 to $20 per ton of concentrate. At 50 million tons annual rate of production, this means ½ to 1 billion dollars. This money involves no generation of compensating profit, but is an expense to maintain ore production and a charge against future profits of the industry.

The question naturally arises as to how this transformation from high-grade to low-grade ore will take place. How will such a complex and unpredictable situation finally be resolved? The answer is given by the individual exploratory projects now in operation or those which are definitely on the planning board. Mention has been made of the Pickands-Mather proving plant. In addition, the Reserve Mining Co., a subsidiary of Oglebay Norton & Co., has taken initial steps leading to the construction of a magnetic taconite concentration plant at Beaver Bay, Minn. Final output is expected to be 10 million tons a year. This same company has started operation of a pelletizing pilot plant at Ashland, Ky., to prove the practicability of agglomerating taconite concentrate. Another company reports that flotation can be successfully applied to a lean specular hematite ore from the Republic formation in Michigan.

It is these pioneering projects that are paving the way toward the final answer. As experience is accumulated and success achieved, the magnitude and scope of operations can be increased.

C. D. King, Iron and Steel Div. Chairman; Paul G. Hoffman, Speaker; and AIME President L. E. Young, just before the ISD Dinner May 25 at Hotel Statler, New York.

Iron and Steel Division Hears Hoffman on "E.C.A. and You"

The largest attendance at an Iron and Steel Division May Dinner, almost 200, turned out to hear Paul G. Hoffman, Administrator of the Economic Cooperation Administration, speak on "The E.C.A. and You." C. D. King, Chairman of the I.S.D. was toastmaster at this very successful dinner, known as the Chairman's Party.

Mr. Hoffman first paid tribute to Dave King and George Wolf for their excellent work in resurveying 400 German plants in order to determine which could better serve our mutual economies by remaining where they are rather than be given to the allies. Not the least of Mr. King's abilities was his diplomacy in convincing the military of this country and the other allies of the advantages of leaving certain plants, previously declared surplus, where they are.

Mr. Hoffman stressed that we should not be deceived into thinking that Russia is trying to spread Communism, rather she is after power. Russia can try to conquer other countries by war or by fifth column tactics. The threat of war must be met by military preparedness and, while Mr. Hoffman felt that the fifteen billion dollars being spent on military defense annually is a serious drain on our economy, it has to be continued as long as the threat of war exists.

The fifth column of the Kremlin prospers on despair. Its success would be tremendously aided by a serious depression. When the Marshall plan was initiated just over two years ago it was specifically directed against poverty, hunger and disease, rather than against any country or doctrine. The fact that it has maintained its original purpose is responsible to a considerable extent for its success in stopping the fifth column in Western Europe.

E.C.A. countries increased their industrial output 14 per cent in 1948 over 1947 and their agricultural output 25 per cent. Mr. Hoffman stressed that E.C.A. was helping improve the capital improvement in these countries and no attempt was being made to improve the standards of living above those which existed in 1938. Conditions are austere but few are hungry. About one billion less dollars will be needed by E.C.A. in the fiscal year 1950 than in 1949, but considerable work must yet be done in removing trade barriers between European nations if they are to become self-supporting by 1952.

The operation of the E.C.A. can best be explained by an example. No money is given to individuals. An English foundryman who needs a piece of equipment from the U.S. makes his request to the English government. After ascertaining that the equipment is needed and that the particular equipment or a satisfactory substitute is not obtainable from one of the soft currency countries, the English government gives him an import license and dollars in exchange for the foundryman's pounds. The pounds stay in the country and are used for capital improvements of public works.

The above policy of buying from soft currency countries wherever possible has saved the U. S. taxpayer large sums.

Institute of Metals Division Fall Meeting

Application forms for hotel accommodations during the fall meeting of the Institute of Metals Division as part of the National Metal Congress have been mailed to members of the Division. Rooms will be assigned first to those making a request on the official blanks. Additional application blanks are available at Institute headquarters if you did not receive one. Be sure to return the room request to the Cleveland address indicated and not to Institute headquarters.

The headquarters hotel for the I.M.D. will be the Allerton. Registration, all technical sessions and the dinner Tuesday evening, October 18 will be held in the headquarters hotel.

All papers to be presented at the fall meeting will appear in the Transactions Section of the JOURNAL OF METALS by October. Although of the record 78 papers, many will be declined or returned for such major revisions that presentation will have to be delayed until the Annual Meeting in New York, February 12-16, 1950, there will undoubtedly be three simultaneous sessions of four papers each on most of the half days starting Monday morning, October 17 and finishing Wednesday afternoon, October 19. The complete program will not be ready for several weeks but papers on both ferrous and nonferrous physical metallurgy, including powder metallurgy, will be scheduled for the Cleveland meeting.

Those who wish to stay at the headquarters hotel are urged to make reservations early. The majority of rooms at the Allerton, both twin and single, are small but reasonable. A few dozen rooms are available at very reasonable rates with running water only. Members are urged to arrange to share twin bedded rooms to avoid being assigned single rooms at considerable distance from the technical sessions.

The subject of this year's ASM Symposium to be held on the Saturday and Sunday preceding the technical sessions of the various societies will be "Thermodynamics in Physical Metallurgy."

Before you leave for the fall meeting, mail your Annual Meeting papers to headquarters. The deadline for all I.M.D. papers and I.S.D. or E.M.D. papers to be preprinted is October 15.

Metal Rolling Is Discussed by Detroit Section, AIME

At the final meeting of the year, May 16, Allen C. Cooley, General Electric Co., spoke before the Detroit Section, AIME, on the rolling of metallic sheet. Mr. Cooley's many years of experience in the reduction of a variety of metals to sheet make him well qualified to discuss rolling mills and their development, theoretical phases of rolling, rolling problems encountered in the laboratory, and the relationship between rolling and the properties of metallic sheet.

Rolling is one of the cheapest methods of metal fabrication, but practical knowledge of the process has far outstripped theoretical knowledge. Mr. Cooley first discussed the theoretical aspects of rolling. Three principal forces—longitudinal, transverse, and vertical—operate during rolling. For simplification it is assumed that the forces are perpendicular to each other, that they are uniform in the plane wherein they operate, and that they remain uniform during operation.

Not all of these assumptions represent a true picture, for the forces are tangential rather than vertical. Furthermore, rolling imposes a variable not accounted for in theoretical considerations. This is forward-and-backward slip. As the stock goes through the rolls it changes in length, and this change in length is associated with a change in peripheral speed.

Under ordinary conditions of rolling there is a point for which the horizontal component of the forward movement of the stock equals that of the roll surface. Back of this point the stock has a forward horizontal component of velocity less than that of the roll surface. In front of it the stock has a greater forward horizontal velocity component than the roll surface. This means that back of the no-slip point there is backward slip of the stock relative to the roll surface; while in front there is forward slip. Rolling is thus a sort of extrusion. The pressure distribution is never uniform over the arc of contact nor across the line of the strip, but is highest at the center.

In speaking of the rolls themselves, Mr. Cooley described the ideal rolls as having the utmost rigidity and operating without bending or flattening. Bending of the rolls produces sheet of nonuniform gage, and undue increase in roll pressure results in flattening of the rolls. These difficulties are avoided somewhat by grinding a crown to the rolls, but this introduces the complication that the crown is not universal. A specific crown is needed for a specific job. In order to increase the roll pressure the trend has been to cut down the area of contact between rolls and stock. The ultimate in this direction has been the development by Rohn of rolls 5/16 in. in diameter, backed up with larger rolls.

Mr. Cooley presented samples of sheet rolled at General Electric Co. under his supervision. He has rolled nearly all of the metals of the periodic table and some 4000 varieties of alloys. He has rolled a welded structure down to 0.000045 in. In this type of work absolute cleanliness is of paramount importance. The stock must be free from dust, grease, or oil.

Mr. Cooley's present interest is in the fabrication of heat resistance sheets for gas turbine applications, which he regards as a challenge to the metallurgical industry.

M. SEMCHYSHEN

Cover Photo Formula

General: Preferably unusual scene.

Subject: Pertaining to metals.

Contrasts: Balance of sharp whites and darks.

Finish: Glossy print.

Taboo: Not too dark.

Example: June or July Cover.

Supplier: You all.

Reasons: Cover Contest.

Eligible: Every one.

Reward:—

First prize: Year's dues to AIME.

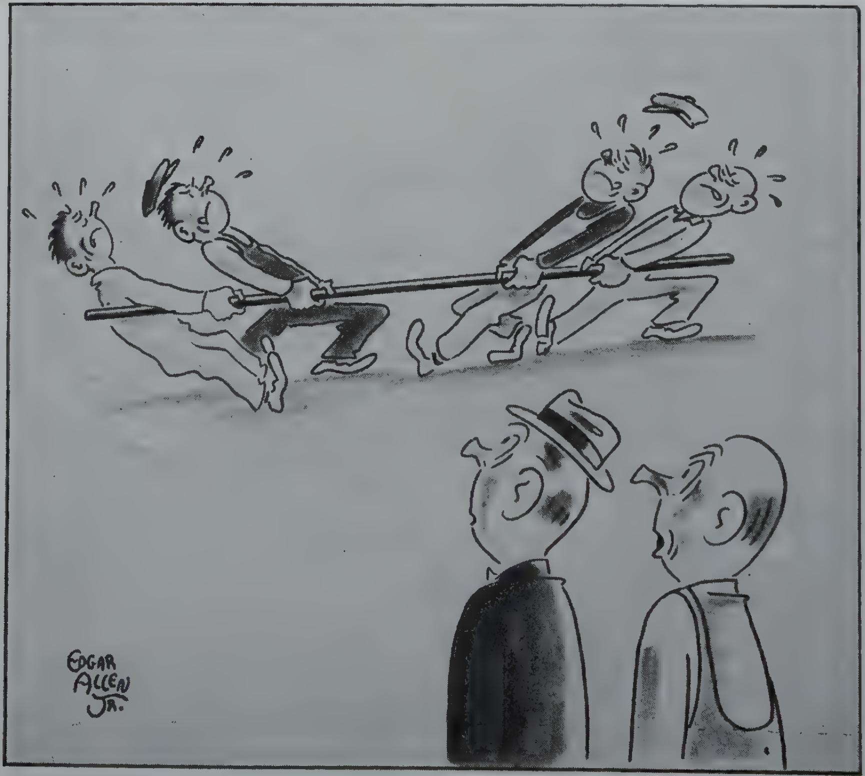
For any photo used: Any copy of IMD's symposium volumes.

Deadline: Sept. 1, 1949.

Comments: Send in your entry!

SMELTS and SMILES

By Edgar Allen, Jr.


EDGAR ALLEN JR.

"Old Donald MacDougal won't buy a tensile testing machine."

ALL INSTITUTE SECTION

APPEARING IN:
- MINING ENGINEERING
- JOURNAL OF METALS
- JOURNAL OF PETROLEUM TECHNOLOGY

Text of Bylaw Amendment to Increase Dues Decided by Board at May 18 Meeting

Numerous matters of more than routine interest engaged the attention of the fourteen Directors who attended the Board meeting at Institute headquarters on May 18. Beginning with an informal luncheon session at 12:30 the meeting continued until 5:40. President Young presided, with the following Directors present: Messrs. Alford, Daveler, Elkins, Fletcher, Head, Kinzel, Kraft, Meyerhoff, Peirce, Phillips, Price, Schumacher, and Sullivan. Sixteen staff members and guests also attended.

The text of proposed amendments to the bylaws was discussed at some length with Edward Holloway, counsel, and the wording as given on another page of this issue was approved. The changes will be voted upon by the Board, according to the bylaws, at a meeting at least two months subsequent to May 18. They will provide for: (1) An increase of $5 in the dues of Members and Associate Members for the years 1950, 1951, and 1952; and for an increase in the dues of Junior Members of $2 for the same years; dues thereafter are subject to a referendum in 1952. (2) A change in the date when dues become payable from Oct. 1 to Jan. 1; members will receive the January, February, and March issues of the journals pending receipt of dues. (3) Elimination of the grade of Junior Foreign Affiliate at the end of 1949. (4) Limiting the term of Student Associateship to the end of the year of graduation, instead of the end of the following year as is the current provision. A special effort will be made to have graduating Student Associates apply immediately for a change of status to Junior Member, though action on their applications will not take effect until the end of the year.

Another matter that aroused considerable interest was the content of the All-Institute Section (Section 2) of the monthly journals. A difference of opinion developed as to whether all personals should be carried in this section, or whether they should be split up into items of particular interest to the three Branches and be published separately in Section 1 of the respective journals. Further discussion of this subject was postponed until the June meeting of the Board.

The advertising situation in the three journals was also reviewed. The mining journal is running somewhat ahead of M&M for the same months of 1948; the metals journal has contracts for $14,000 of the $15,000 income budgeted for the year; and the petroleum journal hopes for $20,000 this year, and at least double that two years hence.

With deep appreciation, especially so at this time, the Board received news of a bequest of £1000, free of English duty, which was left to the AIME in the will of one of its Life Members, William Henry Corbould, who died on March 16.

Reports were made of a preliminary study on the possibility of reducing expenses at AIME headquarters by a merging of certain activities of the four Founder Societies. It is believed that business machines might then be utilized which are not now practical for one society to use. The possibilities are to be investigated further.

A final financial report of the Annual Meeting in San Francisco was made, indicating that registration fees had netted $3708; tickets for various functions, $31,263; and contributions, $13,341. Of the last-named figure, 196 members donated $2381 and 75 manufacturers and companies gave $10,960. Total income was therefore $48,313. Expenses paid for by the San Francisco Section, including $947 returned to New York headquarters to pay a part of the cost of staff assistance at the meeting, amounted to $41,192, leaving a profit to the Section of $7120. This will be invested and used to help in defraying the expenses of future meetings, and to support Section activities. The AIME will have certain expenses not covered in the foregoing figures—chiefly traveling expenses of staff members from New York and Dallas.

Two tellers were appointed from each Branch to count the votes in the referendum for the increase in dues, as follows: Mining, F. X. Corbett and Robert B. Sterns; Metals, David Swan and Thomas A. Read; Petroleum, K. E. Hill and J. I. Laudermilk. The ballots will be counted on June 13, and a report made to the Board at its meeting on June 15. (Results of the balloting show that of the 11,618 ballots mailed, 6,923 valid ballots were returned, 5,403 voting yes, 1,520 voting no. For a tabulation of results see box on page 236.)

Certain changes were voted in the territory of Local Sections in eastern Pennsylvania. The Pennsylvania Anthracite Section's area will be confined to the counties and parts of counties in which there are anthracite mines; the Lehigh Valley Section will hereafter include Cornwall, Lebanon, Lehighton, and Palmerton; and the Philadelphia Section will include some of the area north of Philadelphia heretofore in Lehigh Valley Section territory.

Announcement was made of a survey being made for the U. S. Government to determine which members of various engineering societies have had experience in research and development, and have served in foreign countries. Members of the AIME will shortly receive a questionnaire for this purpose, and its prompt return, filled out as requested, will be appreciated. The work is being done on a contract basis by the ASME, with the co-operation of Engineers' Joint Council.

Ray P. Walters, AIME delegate to the recent World Engineering Congress in Cairo, submitted a detailed report of the meeting to the Board. He felt that technically the Congress was largely a failure. Strong efforts were made by Soviet countries to make political capital out of the meeting, but without much success.

A new Student Chapter, AIME, was recognized at the Institute of Technology, St. Louis University, with Albert J. Frank as Faculty Sponsor. Endorsement was again voted for the Welding Research Council, and its application for $1000 from the Engineering Foundation for its work in 1949. Fred M. Nelson was appointed to the Lucas Medal Committee for a four-year term, and Curtis L. Wilson was reappointed as AIME representative on Engineers' Council for Professional Development for three years. The ECPD annual report was announced as now available. Announcement was made of the United Nations Scientific Conference on the Conservation and Utilization of Resources, to be held at Lake Success, N. Y., Aug. 17 to Sept. 6. The Committee on International Relations of Engineers' Joint Council (of which the AIME is a member) will co-operate.

Immediately before adjournment, a memorial resolution for Sydney Hobart Ball, who died on April 8, was read. It was prepared by Donald M. Liddell.

Proposed Amendments to AIME Bylaws

By direction of the Board of Directors at its meeting on May 18, 1949, the following proposed amendments to the Bylaws of the Institute are published, to be voted on at the July 27, 1949, meeting of the Board, to be held in New York City.

(Add words italicized; delete words in parentheses).

To effect a temporary increase in dues:

Article II, Section 2:

Change second sentence to read as follows: "Dues of Junior Members *shall be* $10 per annum for the first six years of membership as a Junior Member, and thereafter $15; *except that the foregoing dues of Members and Associate Members shall be increased by $5, and of Junior Members by $2, for each of the years 1950, 1951, and 1952 only, unless, as the result of a referendum in 1952, the dues shall be otherwise determined.*"

To effect a change in the date when dues become payable:

Article II, Section 2:

Change fourth and fifth sentences to read as follows: "All dues (for the succeeding calendar year) shall be payable in advance on the first day of (October) *each calendar year*, except as provided in this section, and notice to this effect shall be published in the (September) *December* issues of the monthly journals of the Institute each year. A bill shall be mailed to each member liable for dues (on or before) *prior to* the first day of (October) *January* in each year, stating the amount of the annual dues, date when payable, and the penalty and conditions incident to default in payment."

Article III, Section 2:

In the first sentence, change the word "four" to "three."

To eliminate the class of Junior Foreign Affiliate at the end of 1949:

Article I, Section 1:

Revise first sentence as follows:

"The membership of the Institute shall comprise (seven) *six* classes, namely: 1. Members; 2. Honorary Members; 3. Senior Members; 4. Associate Members; 5. Junior Members; *and* 6. Rocky Mountain Members. (7. Junior Foreign Affiliates.)"

Article I, Section 8:

Delete entire section and renumber succeeding sections.

Article I, Section 10:

Revise first fifteen words as follows:

"Every candidate for election as a Member, Junior Member, *or* Associate Member (or Junior Foreign Affiliate) . . ."

Article I, Section 11:

Revise first sixteen words of second sentence as follows:

"Every person so elected shall become a Member, Junior Member, *or* Associate Member (or Junior Foreign Affiliate) . . ."

Article I, Section 12:

Revise first fourteen words as follows:

"If any person elected a Member, Junior Member, *or* Associate Member (or Junior Foreign Affiliate) . . ."

Article II, Section 1:

Revise first fifteen words as follows:

"Each newly elected Member or Associate Member, and each Junior Member (or Junior Foreign Affiliate) . . ."

Revise last sentence as follows:

"There shall be no initiation fee for Honorary Members *or* Junior Members (or Junior Foreign Affiliates)."

Article III, Section 1:

Revise first thirteen words of second sentence as follows:

"The resignation of a Member, Junior Member, *or* Associate Member (or Junior Foreign Affiliate) . . ."

Article III, Section 2:

In the first sentence, insert the word "or" before "Associate" and delete "or Junior Foreign Affiliate."

To restrict the time during which Student Associates may continue in that status:

Article I, Section 9:

Change the last sentence as follows:

"A Student Associate may remain such until December 31 of the year (following the year) in which he ceases to be in university residence as a student."

Nominees for Institute Officers in 1950

Pursuant to the provisions of the Bylaws as amended, herewith are printed short biographical sketches of the nominees on the official ticket for President, Vice-Presidents, and Directors for the Institute year 1950.

Chairman of the nominating committee of eighteen members is H. Y. Walker.

As provided in the amended Bylaws, 25 Members or Associates may transmit to the Secretary's office "any complete or partial ticket of nominees." If received prior to Sept. 1, a letter ballot will go to all members, containing both the official ticket and any supplementary nominations. If no supplementary nominations are received, according to the amended Bylaws, no letter ballot will be printed and the nominees on the official ticket "shall be declared duly elected at the meeting of the Board of Directors in November."

FOR PRESIDENT AND DIRECTOR

Donald H. McLaughlin, president of the Homestake Mining Co., has served his profession, and the Institute, long and well. A lot of study, traveling, and hard work have filled his 57 years. He graduated from the University of California in 1914, and took his doctorate from Harvard three years later. After two years in the Army, he came back to begin a stellar career. In 1920 he joined Cerro de Pasco and soon after, as chief geologist, was directing the world's second largest geological department.

From 1925 to 1941 he was a professor of mining engineering and mining geology at Harvard, until called to the University of California at Berkeley to serve as dean. During these years, he maintained a consulting practice, serving Homestake and mining organizations throughout the world. Dean McLaughlin became vice-president and director of Cerro de Pasco in 1943. Two years later, he took over the leadership of Homestake. At AIME, Dr. McLaughlin headed the first Committee on Geophysics, and has served on the Mining Geology and Papers and Publications Committees.

FOR VICE-PRESIDENT AND DIRECTOR

Andrew Fletcher is president and treasurer of the St. Joseph Lead Co., and vice-president and treasurer of the AIME. He's a New Yorker, and was 54 last Feb. 6. Mr. Fletcher first worked as a shipyard mechanic in Wilmington, Del., after graduation from Yale's Sheffield Scientific School. Within a short eight years he headed the Fletcher Shipyard in Hoboken, N. J. Maintaining an interest in St. Joe, he became a trustee in 1921, and has been on its executive committee since 1922. Mr. Fletcher is also chairman of the board of the Industrial Hygiene Foundation of America.

Robert W. Thomas, a director of the Institute from 1944 to 1947, and a vice-president since the latter date, has been an engineer for 37 years. He is 63, and general manager for the Nevada Consolidated Copper Co.

Born in St. James, Minn., he graduated from the University of Kansas in 1911. Beginning with the Ray Consolidated Copper Co. as a junior engineer, he moved steadily upward, became superintendent of mines in 1924, and latterly has been general manager at Ray.

FOR DIRECTOR

Harold Decker, 45-year-old petroleum engineer, is assistant manager of the Pan American Production, Pipe Line, and Gas Co. in Houston, Texas. Born in Chicago, he graduated from the University of Oklahoma in 1925. Since then he's been associated with a total of six oil firms, including the Skelly Oil Co. in Tulsa and the Seaboard Oil Co. of Delaware, where he was production superintendent for four years.

Francis B. Foley, superintendent of research for the Midvale Co., Philadelphia, began his metallurgical career with the Company back in 1905, serving there for twelve years. A year of teaching at the University of Minnesota was followed by various activities, including research work with the Bureau of Mines, and collaboration with the late, famed Dr. Henry Marion Howe. In 1926 he rejoined Midvale, assuming his present job. Mr. Foley, a graduate of Girard College, is 61.

Edward C. Meagher, a mining engineer, and president of United Engineering Trustees, Inc., was born in Hoboken, N. J., on Oct. 2, 1897. He attended Columbia University, taking a B.S. degree in 1917, and four years later, an E.M. from the School of Engineering. Mr. Meagher's entire professional career has been spent with the Texas Gulf Sulphur Co. He began there in the sales department in 1921, and is now the firm's treasurer.

Charles Van Ormer Millikan, chief petroleum engineer for the Amerada Petroleum Corp., Tulsa, Okla., has been active in the Petroleum Division since its formation, and was its Chairman in 1930.

Mr. Millikan, 53, is a native Oklahoman and a graduate of Oklahoma A&M. He served in the Navy during World War I, returning to take his M.S. degree from the University of Pittsburgh in 1920. After gathering experience in the Appalachian oil fields for two years, he returned to Oklahoma to join Amerada. He was awarded the Institute's Anthony F. Lucas Gold Medal in 1944.

Howard I. Young, 60, president and general manager of the American Zinc, Lead and Smelting Co. in St. Louis, has spent most of his career in the mining industry. He attended Springfield Business College, joined the firm he now heads in 1908, assuming charge of Missouri mining operations six years later, and becoming president and general manager in 1930. He has been president of the American Mining Congress and the American Zinc Institute.

Gail F. Moulton is a geologist for the Chase National Bank in New York, and a Vice-Chairman of the Petroleum Division, AIME. He is 51, and a graduate of the University of Chicago. Mr. Moulton has 31 years of experience in the petroleum world, formerly headed the petroleum section of the Illinois State Geological Survey, and is a prolific writer in his field.

Division Nominations for 1950

Following are the names of those nominated by the Nominating Committees of the various Divisions for officers for the coming year. In general, the bylaws of each Division provide for additional nominations by petition:

Coal Division: Chairman, Carroll F. Hardy; Chairman-elect, Carroll A. Garner; Executive Committee (until 1953), A. Lee Barrett, Claude P. Heiner, Richard H. Swallow.

Extractive Metallurgy: Chairman, Carleton C. Long; Chairman-elect, Oliver C. Ralston; Secretary, T. D. Jones; Executive Committee (until 1953), Russel B. Caples, A. C. Loonam.

Industrial Minerals: Chairman, Richard M. Foose; Secretary-Treasurer, G. Richards Gwinn; Eastern Vice-chairman, John H. Melvin; Southeastern Vice-chairman, Arthur J. Blair; Rocky Mountain Vice-chairman, Jack C. Pierce; Western Vice-chairman, George D. Dub; Canadian Vice-chairman, Elwood S. Moore; Executive Committee (until 1953), Ian Campbell, Joseph L. Gillson, Stanton Walker.

Institute of Metals: Chairman, Maxwell Gensamer; Senior Vice-chairman, Robert M. Brick; Vice-chairman, Henry L. Burghoff; Secretary, E. O. Kirkendall; Executive Committee (until 1953), William M. Baldwin, Jr., Paul Adam Beck, Alexander Squire.

Iron and Steel: Chairman, H. K. Work; Vice-chairmen, Roy A. Lindgren, Alvin H. Sommer, William A. Steele; Executive Committee (until 1953), Karl L. Fetters, D. L. McBride, George B. McMeans.

Mineral Economics: Chairman, Charles H. Behre, Jr.; Vice-chairmen, Clayton G. Ball, Richard J. Lund, Charles White Merrill; Secretary-Treasurer, Joseph A. Corgan; Executive Committee (until 1953), Simon D. Strauss, W. W. Mein, Jr., and R. J. Bradley.

Mineral Industry Education: Chairman, Allison Butts; Vice-chairman, Harry H. Power; Executive Committee (until 1953) James D. Forrester, John R. Spielman, J. W. Stewart.

Minerals Beneficiation: Chairman, Grover J. Holt; Associate Chairman, Raymond E. Byler; Regional Vice-chairmen, Edwin H. Crabtree, Jr., Donald W. Scott; Secretary-Treasurer, Will Mitchell, Jr.; Committee Chairmen—Membership, James A. Barr, Jr.; Papers and Publications, M. D. Hassialis; Program, Raymond E. Byler; Education, S. R. B. Cooke; Concentration, Milton LeBaron; Materials Handling, Stanley D. Michaelson; Crushing and Grinding, Fred C. Bond; Solids-Fluids Separation, F. W. McQuiston, Jr.; Operating Control, F. M. Lewis; Solution & Precipitation, Max W. Bowen; Pyrolysis & Agglomeration, Fred D. DeVaney; Richards Award, E. H. Rose, J. F. Myers; Symposium, A. F. Taggart; Nominating, T. B. Counselman.

Petroleum Division: Chairman, John E. Sherborne; Vice-chairman, Paul Andrews; Executive Committee (until 1953), R. W. French, R. C. Earlougher, John R. McMillan.

Results of the Balloting on Increased Dues

Ballots mailed 11,618

Branch	Yes	No	Percent Yes
Mining	2,258	651	77.5
Metals	1,339	366	78.5
Petroleum	1,306	357	78.5
Other	500	146	77.4
Total	5,403	1,520	78.2

What Went on at Recent Local Section Meetings

SECTION	DATE	ATTENDANCE	SPEAKER, AFFILIATION, AND SUBJECT
Arizona, Morenci Subsection	April 12	33	William Distler, "Proverbs for the Young Engineer." J. E. Skipper on the Morenci power plant and the future of the gas turbine.
Black Hills	May 6	90	Joint meeting, Woman's Auxiliary and Student Associates from S. Dak. School of Mines & Technology. J. V. N. Dorr, The Dorr Co., on early development of Dorr equipment in Black Hills.
Boston	May 2	72	Joint meeting with Woman's Auxiliary. Seville R. Davis, American editor, Christian Science Monitor. "Analysis of Postwar U. S. Foreign Policy in Europe."
Carlsbad Potash	May 12	52	J. Harlan Johnson, Colorado School of Mines, comments on International Geological Congress.
Columbia	May 14	120	Third Annual Northwest Industrial Minerals Conference.
Cleveland	May 12	....	Harry A. Schwartz, National Malleable & Steel Castings Co. "Correlation of Physical Properties with Chemical Composition in Low Alloy Cast Steels."
Detroit	May 16	....	A. C. Cooley, General Electric Research Lab. on metallic sheet.
Mid-Continent	April 9	100	John T. Hayward, Barnsdall Oil Co., on unique design for offshore drilling structures.
Montana	April 26	130	R. H. Townsend, Anaconda Copper Mining Co., on ferromanganese operations.
Montana	May 27	....	Joint meeting with Last Chance Gulch Mining Assn. W. R. Wade, Rainbow Mining Co., on the Marysville district.
New York	May 18	75	J. D. Doherty, office of synthetic liquid fuels, Bureau of Mines, "Synthetic Oil—Security for the Future."
Ohio Valley	May 18	22	Film, "Ohio and its Mineral Resources." Progress report on AIME Mid-Year Meeting.
Pennsylvania-Anthracite	April 22	195	S. H. Ash, chief, safety branch, Bureau of Mines, on anthracite flood-prevention project.
St. Louis	April 22	86	J. G. Evans, Gardner-Denver Co., on mining experiences in South Africa.
Utah	April 21	44	Clayton Wolf, Univ. of Utah engineering experiment station, on use of noncoking coal in metallurgical processes.

Engineering Index Volumes Wanted

Copies of the bound volumes of Engineering Index from 1928 to date are needed by various libraries and organizations, both in this country and abroad. If you have volumes that you do not need, please get in touch with Ralph H. Phelps, director, Engineering Societies Library, 29 West 39th St., New York City 18.

The Drift of Things

The Referendum

One may look at the results of the referendum on increased dues in one of two ways. It may be considered reassuring that three fifths of the AIME members qualified to vote returned their ballots, and that four out of five of them voted for the increase although it definitely meant an out-of-pocket expense to them for the next three years. Also, it is good to know that all three Branches—Mining, Metals, and Petroleum—show the same four-to-one ratio in favor of increased dues.

But speaking as Secretary of the AIME, we choose to look at the vote with something less than complete satisfaction. Serious thought should, we believe, be given to the fact that some 1500 members do not seem to think the service they are getting, or are likely to get in the future, is worth more than the $15 or the $10 that they have been paying for the the last three decades, in spite of the acknowledged greatly increased cost of practically everything. Also, in spite of what we are assured was an excellent percentage of returns, the fact remains that about two fifths of the Institute membership was too busy or not sufficiently interested to vote at all. Although they were not actively against the proposed increase in dues, they did not support it, even to the extent of making a couple of check marks, signing their name, and dropping a postal card into the nearest letter box.

Unquestionably, much more work must be done in making the Institute what its members want it to be. Most tangible of the services that can be improved, of course, are the publications—monthly journals and books, and the meetings—annual, mid-year, Divisional, and Local Section. But also the officers, Directors, and headquarters staff of the AIME must get closer to the members, must think about and talk over each others' problems, and must discover what new fields offer promise of better service to members.

We feel that a good start has been made, but far more must be accomplished in the next three years. At that time another referendum will be taken, and it is hoped that in the meantime it will be possible to increase advertising income, the sale of books, and the number of members sufficiently to make a return to the current dues schedule possible. Other avenues of income may also be developed, or methods of cost cutting devised, so that the standard of services to AIME members can be maintained, or even expanded, even though prices will not have retracted to the prewar level.

Those of us whose responsibility it is to run the Institute must, in the next three years, justify our stewardship. But the AIME is a co-operative enterprise, and the best results can only be secured if the members at large also will aid in every way they can to secure and prepare more and better papers, and to hold more interesting meetings.

Reviewing the First Six Months of the New Journals

At the Board meeting last September, approval was given for the three new journals whose publication began with the issues for January 1949. According to the minutes of that meeting, "Section one of each of the journals would be devoted to advertising and editorial matter of specific interest to the individual Branch and to be secured, edited, and wholly under the control of the Branch council or editor. Section two would be common to all three journals and would consist of feature articles, shorter material, and professional news prepared and edited at Institute headquarters but of a wide interest and character to be approved by each Branch council or editor. Section three would consist of technical papers of interest to the respective Branches, as now published in the Technologys." It was planned at that time that Section one of the three journals would average about as follows, as to number of pages per issue: Mining, 48; Metals, 16; Petroleum, 32. Section two would average about 24 pages for each of the journals. The actual number of pages for the first half year has not been greatly different from these estimates. Section three averages were: mining, 37; metals, 65; and petroleum, 28 pages.

At the May Board meeting, proposals of the three editors were advanced to reduce the size of Section two (the All-Institute section) to an average of about eight pages per month. This would involve elimination of all personals and obituaries from the All-Institute section and assigning them to Section one, according to Branches. The material still to be put in Section two would include reports of Board meetings, a page or two of the Drift of Things, the calendar of coming meetings, a summary table of Local Section meetings, a list of the titles of all technical papers published in the three journals with a definitive sentence as to the subject covered, a list of those applying for membership (though this might be split according to Branches and published in Section one), publicity regarding Annual and Mid-Year meetings, short book reviews of general interest, brief Woman's Auxiliary news, and short biographies of the President and new Directors each year. An occasional feature article might be used if space permitted. The annual reviews, which bulked so large in the March issue, would be split up and published only in the journal covering their specific field.

Those present at the Board meeting were, of course, the more mature members of the Institute, and they expressed considerable opposition to further condensation of the All-Institute section, particularly as to elimination of the personals. Their interests are naturally broad, and their friends and acquaintances fall into all three Branches. They pointed out that the present scheme of publishing has met with almost unanimous approval and they urged that no further change be made until at least one full year has been completed. In the meantime, however, the editors will welcome comment from members as to the type of material they like in the present journals, and what classes, if any, they do not find of interest.—*E. H. R.*

Mid-Year Meeting News

Governor Lausche

A Message from the Governor of Ohio

"I am happy to learn that the 1949 Mid-Year meeting of your Institute is going to be held in Columbus, Ohio, the week of September 25th.

"It is a pleasure and privilege to welcome the officers, members, and guests of your organization to our Capital City.

"It might interest you to know that Ohio is one of the leading mineral producing states, with coal, oil and gas, limestone, clay, glass sand, salt, gypsum, and numerous other minerals all mined within our borders. Ohio is also rich in scenic attractions and historical lore.

"I am, therefore, pleased that you have chosen our state as the place for your meeting, and I know that in the friendly atmosphere of Columbus your sessions will be both enjoyable and educational."

FRANK J. LAUSCHE,
Governor, State of Ohio

A Message to Members:—

The Mid-Year Meeting of the Institute, scheduled for Columbus during the week of September 25, 1949, will afford a splendid opportunity for members and their friends to participate in an outstanding technical program. There will be papers and conferences on current problems of industry, management, and technical education in the mineral technology field; and inspection trips to some of the nation's finest industrial and research plants.

A most cordial welcome has been extended to all Institute members to visit Columbus on this occasion by the Governor of Ohio, the Mayor of Columbus, and the officers of the great industrial, research, and educational institutions, as well as by the Ohio Valley Section of the Institute.

The AIME Directors hold their September meeting at Columbus on September 27, with representatives of the Sections and Divisions present to discuss the program and progress of the Institute. The social functions planned for the ladies, as well as the members, will add greatly to the pleasure of the meeting. Adequate hotel accommodations are assured.

On Friday, Sept. 30, the Southern Ohio Section of the Open-hearth Committee, AIME, will begin a two-day meeting, also at Columbus, so many members will find that their time can be profitably occupied for the entire week.

The Mid-Year Meeting presents a splendid opportunity for many who could not be at San Francisco to participate in an important national gathering of mining and petroleum engineers and metallurgists for which most diversified programs have been arranged.

L. E. YOUNG,
President, AIME

Women Plan Gala Meeting

Several hundred women from all parts of the country will arrive in Columbus on September 24 with their engineer spouses, and the Columbus Section of the Woman's Auxiliary, AIME, will play the gracious hostess during the Mid-Year Meeting. Mrs. Elmer R. Kaiser, 2406 Brixton Rd., Columbus, general chairman of the ladies' committee, has been busy arranging the entertainment.

Following the All-Institute luncheon on Monday afternoon, the ladies have scheduled a sightseeing trip through Ohio's capital city, winding up the day with a tea at the home of the president of Ohio State University. Tuesday will feature a visit to the Heisey Glass Co. at Newark, Ohio, lunch at the Granville Inn, and then a tour of the plant where the new Lustron houses are made. A fashion show and luncheon at the Maramor, famed Columbus restaurant, will be Wednesday's high spot, but the ladies will go higher that evening to dine with their husbands atop a forty-story skyscraper in the heart of the city. A romantic nocturnal visit to the local observatory is planned for later that night, with a planetary display on the agenda.

Columbus's Mayor Welcomes the AIME

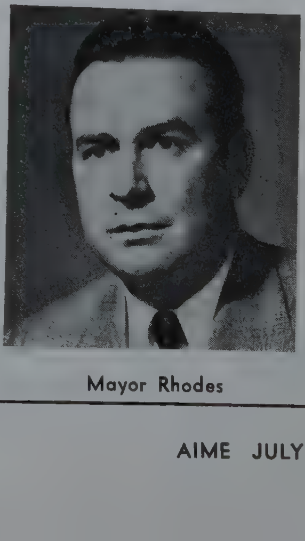
"Columbus appreciates the honor and privilege of playing host to the American Institute of Mining and Metallurgical Engineers.

"Columbus is the heart of a great state. It is Ohio's capital city. Meeting in Columbus is traditional, for hundreds of conventions are held in our city each year. There are many fine hotels and restaurants to accommodate visitors.

"It's easy to come to Columbus. Our city is conveniently accessible to the country's principal metropolitan centers by air, rail or highway. One half ot the nation's population resides within a 500 mile radius of Columbus.

"We urge you to attend your mid-year meeting. We know you'll enjoy your stay, although brief, in Columbus."

JAMES A. RHODES, Mayor,
City of Columbus, Ohio



Mayor Rhodes

Part of the broad and beautiful campus of Ohio State University in Columbus, where 2600 educators serve a student body numbering 24,000. This is but one of the 54 colleges and universities which make Ohio one of the nation's best educated sections.

Ohio the Great

Ohio, native state of eight Presidents, seventh in rank among the states producing industrial minerals, leading the world in rubber, steel, and clay products, will play host to the AIME in September. Ohio is the Iroquois word for "great." Ohio's great, and our Mid-Year Meeting will be the same.

There'll be plenty of vacationing to do in the Buckeye State when technical sessions are over. Five hundred and thirty miles of rivers and a 230-mile frontage on Lake Erie furnish scenic beauty, and direct commerce routes with the world. Great steel and other industries along the waterways move observers to label Ohio "The Ruhr of America." A storehouse of minerals lies below the fertile farmland, and 117,000 businesses operate atop it. Columbus, the state capital, with the Scioto River winding like a ribbon through her hair, awaits Mr. and Mrs. AIME.

Ohio is mostly farmland, but oil derricks hold up the sky, and the world's largest sandstone quarry (right) is in S. Amherst. The soil over which the pioneers traveled in 1788 now yields a rich harvest of food, and the minerals vital to commerce.

In the Buckeye State, heavy industry joins with ante-bellum charm, providing lifeblood for the world's commerce, and pleasure for visitors. Up and down the Ohio and Muskingum rivers flow the iron ore, coal, and limestone which keep the blast furnaces going.

Among the Student Associates

Mackay Grads Feted by Reno Section

The twenty-two members of the graduating class at the University of Nevada's Mackay School of Mines were the honored guests, on May 5, at a luncheon meeting of the Reno Section, AIME. Chairman A. C. Johnson presided, and called upon Jay A. Carpenter to introduce the graduates individually.

All twenty-two graduates are Student Associates of the AIME, credit for this fine record going to H. B. Chessher, Sr., chairman of the Reno Section's membership committee. Alberto Barrios, retiring president of the Crucible Club, addressed the gathering, and then went on to provide fine entertainment, singing and accompanying himself on the guitar.

Lehigh University

The Howard Eckfeldt Society met for the last time this year on May 19, viewing a film and electing officers for next year. The film was a Phelps Dodge Copper Co. presentation, showing mining and milling operations at Bisbee, Ariz.

The men who'll pilot the Society through the next year are: President Robert L. Smith; Vice-President Robert A. Limons; Treasurer Joseph A. Holmes; and Robert E. Nolan, Jr., Secretary.—*Robert E. Nolan, Jr., Secretary.*

Calendar of Coming Meetings

JULY

7 **Reno Branch, Nevada Section, AIME.**
9-23 Fourth Empire Mining and Metallurgical Congress, Great Britain.
13 **El Paso Metals Section, AIME.**
13 **Southwestern New Mexico Section, AIME.**
13-15 American Society of Civil Engineers, summer convention, Mexico City.
15-24 Pan-American Engineering Congress, Rio de Janeiro.
19 **Gulf Coast Section, AIME.**
25 **Alaska Section, AIME.**

AUGUST

23-26 AIEE Pacific general meeting, Fairmont Hotel, San Francisco.
26-Sept. 10 Engineering & Mining Exhibition, London, England.
Aug. 17-Sept. 6 United Nations Scientific Conference on the Conservation and Utilization of Resources, Lake Success, N. Y.

SEPTEMBER

25-28 American Mining Congress, Metal Mining Convention, Hotel Davenport, Spokane, Wash.
25-28 **Mid-year Meeting, AIME, Neil House, Columbus, Ohio.**
29-30 ASME, fall meeting, Erie, Pa.
29-Oct. 1 Colorado School of Mines, 75th anniversary celebration.
Sept. 30-Oct. 1 Southern Ohio Section of Open Hearth Committee, AIME, fall meeting, Deshler-Wallick Hotel, Columbus.

OCTOBER

5-7 **Petroleum Branch, AIME, fall meeting, Plaza Hotel, San Antonio, Texas.**
13-14 Texas Mid-Continent Oil and Gas Association, annual meeting, Rice Hotel, Houston.
14 **Eastern Section, Open Hearth Committee, Iron and Steel Division, annual all-day fall meeting, Warwick Hotel, Philadelphia.**
14 **Southwestern Section, Open Hearth Committee, Iron and Steel Division, Kansas City, Mo.**
17-19 **Institute of Metals Division, AIME, fall meeting, Allerton Hotel, Cleveland.**
17-23 AIEE, 1949 Mid-West meeting, Netherland Plaza, Cincinnati.
20-21 **Petroleum Branch, AIME, Elks Club, Los Angeles.**
24-28 Thirty-seventh National Safety Congress and Exposition, Chicago.
26-27 **Joint Fuels Conference, ASME-AIME, French Lick Springs Hotel, French Lick, Ind.**
28 **Pittsburgh Section of Open Hearth Committee and Pittsburgh Section, AIME, annual fall meeting, William Penn Hotel, Pittsburgh.**
28-29 ECPD, annual meeting, Edgewater Beach Hotel, Chicago.

NOVEMBER

1-5 Pacific Chemical Exposition, California Section, American Chemical Society, San Francisco Civic Auditorium.
2-4 American Society of Civil Engineers, fall meeting, Washington, D. C.
7-10 AIChE, annual meeting, Pittsburgh, Pa.
9-11 **Industrial Minerals Division, AIME, Tampa, Fla.**
12-14 Geological Society of America, annual meeting, Hotel Cortez, El Paso.

DECEMBER

7 American Mining Congress, Annual Business Meeting, New York City.
8-10 **Seventh Annual Conference, Electric Furnace Steel Committee, Iron and Steel Division, AIME, Hotel William Penn, Pittsburgh.**

JANUARY 1950

18-20 American Society of Civil Engineers, annual meeting, New York.
30 AIEE, winter meeting, New York.

FEBRUARY 1950

10 **Southwestern Section, Open Hearth Steel Committee, Iron and Steel Division, St. Louis, Mo.**
12-16 **Annual Meeting, AIME, Statler (Pennsylvania) Hotel, New York City.**

APRIL 1950

2-4 **Annual conference, National Open Hearth Committee, Iron and Steel Division, Cleveland, Ohio.**
10-12 **Open Hearth Conference, and Blast Furnace, Coke Oven and Raw Materials Conference, Netherland Plaza Hotel, Cincinnati.**
19-21 American Society of Civil Engineers, spring meeting, Los Angeles.

DECEMBER 1950

7-9 **Eighth Annual Conference, Electric Furnace Steel Committee, Iron and Steel Division, AIME, Hotel William Penn, Pittsburgh.**

University of Nevada

At the April 27 meeting of the Mackay School of Mines' Crucible Club, Mr. Zadra, of the Rare Metals Station of the Bureau of Mines in Reno, presented, for the first time, his paper on the electrolytic deposition of gold from activated charcoal. The students found the session most interesting and educational.

The Club then went on to elect officers for next year. Fred Muller is the new President, with John Cunningham, Walter Johnson, and Harry Varischetti serving as Vice President, Secretary and Treasurer, respectively.

Club members can now look back on a highly successful semester of activities. W. E. Wrather, USGS head, and former AIME President, addressed the group in February, and the March gathering heard a report from President Barrios on the AIME Annual Meeting in San Francisco. Since the first of the year, the Club has sponsored several movies for students and the public, including films on the natural resources of Nevada, block caving methods at the Emma Nevada mine in Kimberly, and one on the various types of Marion shovel in operation. The alumni paper, "The Mackay Miner," was published during the first week of May, and mailed to alumni, former students, and friends of the club.—*Walter B. Johnson, Secretary.*

University of New Hampshire

An aerial field trip, aimed at studying the glacial features of Illinois, and made last summer by the students of the University of Illinois, was described to University of New Hampshire Student Chapter members at their regular April meeting. D. H. Chapman, associate professor of geology at New Hampshire, described the airborne venture, which he had directed, and illustrated his talk with excellent color slides.

New officers for the coming year were elected at another April meeting. New Chapter President Lawrence W. Wakefield is ably supported by Vice-President Carl A. Lein; Secretary-Treasurer Mrs. Isabelle A. Kerdack; and Corresponding Secretary Livingston Chase.—*Marston Chase, Corresponding Secretary.*

News of AIME Members

Marshall B. Ames was transferred early in January by the Corps of Engineers from the Alaska district to the Walla Walla, Wash., district.

E. W. K. Andrau expects to do considerable research and prospecting for uranium in the Rocky Mountain area. The E. W. K. Andrau Enterprises has opened an office in Lander, Wyo., where it is expected that Dr. Andrau will in the near future do considerable geological work in addition to some drilling this year.

Murray R. Arrowsmith can be found in Kimberly, Nev., where he is working as a geologist for the Consolidated Coppermines Corp.

L. J. Barraclough, who is reached at the Disergarh Post Office, Burdwan District, W. Bengal, India, has been elected president of the Mining, Geological and Metallurgical Institute of India.

Ralph C. Beerbower, former district manager of the Goodman Mfg. Co., has taken the post of assistant to the superintendent of the H. C. Frick Coke Co., West Leisenring, Pa.

Chas. H. Behre, Jr., returned late in April from Rangoon, where, as representative of Behre Dolbear & Co., mineral advisers to the Burma Union Government, he had been discussing general plans for mineral development. The trip involved a round-the-world flight. He left to resume field work in Mexico on May 15, and on June 7 received an honorary degree of Doctor of Science from Franklin and Marshall College, Lancaster, Pa.

Josef W. J. Bercher is assistant division manager of the Texas Eastern Transmission Corp., P. O. Box 32, Downingtown, Pa.

Bernard Beringer, former assistant general manager of Randfontein Estates G. M. Co., is now mine manager of Palmiet Chrome Mines Ltd., P. O. Matooster, Rustenburg, Transvaal, S. Africa.

Charles W. Campbell has changed from the engineering department to the production department of Mina Tiro General, Charcas, S.L.P., Mexico, and is now shift boss at the property.

K. P. Campbell, working for the American Smelting and Refining Co., has been transferred from Chihuahua, Mexico, to the Company's general engineering department at El Paso, Texas.

William J. Coulter, Jr., is working as a miner for the Bunker Hill and Sullivan Mining and Concentrating Co. His address is P. O. Box 353, Kellogg, Idaho.

Orville R. Dahlberg, since graduating from the University of Wisconsin, has been employed by the Oliver Iron Mining Co. as a mining engineer on a training program, intended to acquaint future operators and engineers of the company with the various operations that comprise the company as a whole. Periods of varying lengths are spent under the different departments, such as engineering, special exploration, budget control, shops, grading, sampling and safety.

Rene Engel

Rene Engel, geologist and chemical engineer, recently became associated with **Florent H. Bailly,** petroleum engineer and geologist, founder and president of Oil Properties Consultants (geology and engineering) and Petroleum Engineering Associates (laboratory studies), both of Pasadena, Calif. Under the names of Petroleum Industry Consultants, Cuba, and Petroleum Industry Consultants, Venezuela, this association is extending its services from branches located respectively in Havana and Caracas. Dr. Engel still retains his post as curator of mineralogy and petrology at the Los Angeles County Museum.

Paul B. Entrekin

Paul B. Entrekin, former vice-president and manager of Bethlehem Chile Iron Mines, became manager of the Bethlehem Steel Company's mining division in May. A Lehigh graduate, Mr. Entrekin started with Bethlehem in 1925 as an engineer in the mining department. He was transferred to the Cornwall mine in 1926 to service successively as plant engineer, mine superintendent, and assistant manager. In 1941 he went to El Tofo, Chile, as mine superintendent and assistant manager of Bethlehem Chile, a Bethlehem Steel subsidiary, becoming manager and vice-president in 1944.

Lloyd L. Connell writes that his connection with the Stoker Manufacturers Association terminated on April 1 and that he is now with the Diamond Chain Co., Indianapolis, Ind. Following completion of his current period of training, he expects to be assigned to a territory as sales representative.

John M. Davis can be reached at the Western Machinery Co., 231 Healey Bldg., Atlanta 3, Ga. He had worked for the Phosphate Mining Co. in Florida.

Joseph M. Downey, formerly metallurgist at the Leadville milling unit of the American Smelting and Refining Co., is now a miner at the Kokomo Victory mine of the Company.

John R. Drenan has been appointed superintendent of the fluorspar division of the United States Coal and Coke Co. at Mexico, Ky., effective May 1.

L. E. Young, guest of honor at St. Louis Section—Missouri School of Mines Student Chapter banquet on May 14, with St. Louis Chairman Herbert Franke and Mrs. Franke.

L. E. Young, President of the AIME, spoke before the Missouri School of Mines Student Chapter on May 14. He discussed briefly technical assistance to foreign countries and then spoke at length on potash mining, illustrating his talk with slides and maps. The Student Chapter reports that Dr. Young's wide experience, coupled with natural speaking ability, gave a comprehensive picture of this type of mining, and members of the Chapter feel that this was the most important meeting in the history of the Chapter.

Gilbert C. Davis acted as manager of the Stag Canon branch of the Phelps Dodge Corp. from 1929 to 1939 when he was transferred to the Morenci branch where he was in charge of operations until 1946. At that time he was transferred to Douglas, Ariz., as director of labor relations, and continued in that capacity until his retirement on March 1. To go back thirty odd years, Mr. Davis went from Denver to the Rock Springs district to open a new coal mine for the Colony Coal Co. at Dines, Wyo., and was in charge of operations until 1927 when he moved to Mt. Harris, Colo., and for the next two years was general manager of the Colorado & Utah Coal Co. as well as the Colony Coal Co.

G. Dessau, an Italian mining engineer, who, after having been taken to India as a prisoner of war, was later allowed to work there and was in charge of the geophysical section of the Geological Survey of India from the start of that section, has now returned to Italy. He has again taken up service with the Italian Colonial Administration and has been attached to the Geological Survey of Italy.

O. J. Egleston has retired as vice-president and consulting engineer for the U. S. Smelting Refining and Mining Co. after 46 years with the Company and its predecessor. **A. B. Marquand** has also retired as vice-president and manager of metal sales after 31 years with the concern.

Andrew Fletcher, president of the St. Joseph Lead Co., and vice-president and treasurer of the AIME, delivered the commencement address at the Missouri School of Mines and Metallurgy, Rolla, on May 31. At the same time the University of Missouri conferred upon him the degree of Doctor of Engineering.

Milton H. Fies has been appointed a consulting engineer to the Office of Synthetic Liquid Fuels, Bureau of Mines. He will continue as part time manager of coal operations for the Alabama Power Co. Under Dr. Fies's supervision, the Alabama Power Co. is collaborating with the Bureau of Mines in a second field-scale experiment in the underground gasification of coal at Gorgas, Ala. A seam of coal there recently was ignited under carefully controlled conditions to determine the feasibility of using the gases thus produced as a source of fuel for boilers and as a raw material for making synthetic oil and gasoline.

Glenn H. Fritz left his job with the Anaconda Copper Mining Co. last December and is now employed by the Joy Mfg. Co. as a sales trainee.

J. Porter Gardner, Jr., formerly field ventilation engineer for the Joy Mfg. Co. at their New Philadelphia, Ohio, plant, was transferred on Jan. 1 to Joy's Chicago district sales office as sales representative specializing in fan sales.

Dion L. Gardner has moved his consulting office in geology and engineering to 1815 W. Chapman, Orange, Calif., c/o Association Laboratories. His office was in Santa Ana.

Fred E. Gray is living at the Asbury Apartment Hotel, 2505 W. 6th St., Los Angeles 5. He is assistant general manager of the Coronado Copper and Zinc Co.

I. G. Grossman, formerly at the department of geology at the University of North Dakota, is now with the petroleum economics branch of the Bureau of Mines in Washington, D. C.

Julius B. Haffner has been elected second vice-president of the Bunker Hill and Sullivan Mining and Concentrating Co. and **Donald H. McLaughlin** becomes a director of the Company.

Alfred S. Harvey, since graduation from the Missouri School of Mines and Metallurgy last June, has been quarry foreman for the U. S. Gypsum Co. at the Fort Dodge plant. He can be reached at 1111 S. 18th St., Fort Dodge, Iowa.

John S. Hemphill is now associated with C. A. Venezolana de Cementos, Caracas, Venezuela.

James M. Hill, formerly geologist with the Union Carbide and Carbon Co., has opened his office as a consulting mining geologist at 182 N. Court St., Tucson, Ariz.

Arne Hofseth is one of two Norwegian technicians who will spend six months in the United States studying mining methods and equipment which might be adapted for use in mine operations in Norway. They are particularly interested in modern methods of open pit mining, equipment used for milling and transporting ore, and American methods of pelletizing iron ore. The Bureau of Mines is co-operating with ECA's technical assistance division in arranging the studies.

John A. Johnson is supervisory engineer of the Duluth office of the Health and Safety Division of the Bureau of Mines.

Werner O. Joseph, upon graduation from the University of Chile, became assistant mill foreman of Cia. Minera Punitaqui in Ovalle, Chile.

Francis R. Joubin, for twelve years on the exploration and mine staff of Pioneer Gold Mines of British Columbia, and more recently engaged as their Eastern Canada representative, has been granted a leave of absence allowing him to assume charge of exploration projects for other companies. He will be retained as Pioneer's eastern adviser on exploration, and will continue to make his headquarters at the Pioneer company's offices at 510 Concourse Bldg., 100 Adelaide St., W., Toronto, Ont.

Nicholas N. Kohanowski, formerly with Cie. Aramayo de Mines en Bolivie, has returned to the States and accepted the post of assistant professor in the department of geology at the University of North Dakota, Grand Forks.

Emerson C. Lane, has gone to San Juancito, Honduras, as metallurgist for the New York and Honduras Rosario Mining Co.

H. F. Lynn informs us that he is now associated with the Southwestern Engineering Co., 4800 Santa Fe Ave., Los Angeles 11, Calif. He had been with the Western-Knapp Engineering Co.

Mark C. Malamphy, consulting geophysicist, formerly of Little Rock, Ark., and W. G. Garlick, consulting geologist of Lusaka, Northern Rhodesia, have incorporated under the firm name of Mark C. Malamphy & Co., Ltd., consulting geologists and geophysicists with offices in Mufulira, N. Rhodesia. Mr. Malamphy has been engaged in geophysical work in the United States, Canada, and South America since 1926, and in the Union of South Africa, the Rhodesias, and the Belgian Congo since 1946. Mr. Garlick has been doing geological work in South and Central Africa for about twenry years. The new firm's address is P. O. Box 209, Mufulira, N. Rhodesia.

Charles A. Marr is working for the St. Joseph Lead Co. at Bonne Terre, Mo., as a mining engineer.

Robert R. Mates, since school days at the University of Utah, has been employed by the Kennecott Copper Corp., Arthur, Utah, as a research engineer and by the Bureau of Mines at Salt Lake City as a metallurgist.

J. L. McCluggage is a mining engineer with the Kamunting Tin Dredging Co., Taiping, Perak, Malaya. He had held the same post with the Rawang Tin Fields, Ltd.

C. E. McManus has been appointed assistant to the general manager of the Minnesota mines of The M. A. Hanna Co.

Wilson D. Michell, after spending two years in French Morocco as geologist for the Societe Nord-Africaine du Plomb, engaged in the exploration and development of lead-zinc deposits in the northeastern part of that country, is now working as a geologist for the Resurrection Mining Co. at Leadville, Colo.

Arnold H. Miller, consulting engineer of New York City, has been in Sinaloa, Mexico, on professional work.

F. Stuart Miller has 50 S. Remington Rd., Columbus, Ohio, as a permanent address, but right now he is in Kuala Lumpur, Malaya, as assistant manager of the Pacific Tin Consolidated Corp.

James T. McDonald is working for the Bunker Hill and Sullivan Mining and Concentrating Co. at Kellogg, Idaho, as a surveyor.

Clarence O. Mittendorf is on an ECA mission to Turkey. He can be reached at Ankara in care of the American Embassy.

C. C. Huston

C. C. Huston, former head of the exploration department of Macassa Mines Ltd., has resigned from that post to enter into private consulting practice. His address is 1001-85 Richmond St., W., Toronto.

R. M. Mahoney

R. M. Mahoney is continuing his activities with the United States Vanadium Corp. as manager of industrial relations but has moved his headquarters to Bishop, Calif.

Ivan L. Nichols is working as a metallurgical trainee for the U. S. Smelting Refining and Mining Co. at Midvale, Utah. He had been with the Pacific Bridge Co. in Park City.

C. M. Peters, who left his post as geologist and chief engineer at the Neptune Gold Mining Co. after completing one year, is now working out of San Francisco for the firm of Dames and Moore, who do foundation engineering and soil mechanics. He is their geologist and job engineer on work that consists of testing the soil and formation for foundations of buildings, bridges, and dams.

R. G. Ponsford recently became a member of the Ponsford-Moos Equipment Co., El Paso. He was formerly with the Mine and Smelter Supply Co. and takes to his new post many years of experience specializing in mining, industrial, and shop equipment.

Burton M. Reynolds was appointed mining engineer, Rolla branch, Mining Division, Bureau of Mines, last November, going there from Idaho. He was formerly with the Bureau in College Park, Md. His present assignment is in Little Rock, Ark.

T. A. Rickard, of Victoria, B. C., has been appointed an honorary fellow of the Imperial College of Sciences. Most octogenarians are satisfied to sit back and let the world carry on by itself, but not Dr. Rickard. Last year he published "Historic Backgrounds of British Columbia," and recently his new book, "Autumn Leaves," has appeared.

Ernest R. Rodriguez is working as a mining engineer with the safety branch of the Bureau of Mines with headquarters at Phoenix, Ariz.

John A. Roos is working for the Baroid Sales Division of the National Lead Co. as a mining engineer. His address is Box 265, Belle Fourche, S. Dak.

John G. Reilly resigned as general manager of the Bayard, N. Mex., department of the United States Smelting Refining and Mining Co. on May 21. He will enter the consulting field and will continue to maintain his residence at 407 College Ave., Silver City, N. Mex. Mr. Reilly formerly was managing director of Cia. Real del Monte y Pachuca at Pachuca, Hidalgo, Mexico.

Joseph T. Roe is construction engineer for Dent and Russell Inc., Dantors division, mining, milling, and manufacturing, in Maupin, Oreg.

H. L. Roscoe, vice-president and general manager of Noranda Mines Ltd., who had been resident in Noranda since the beginning of operations, has moved to the head office in Toronto. **R. V. Porritt,** who has served the Company since 1926, for the last ten years as assistant general manager, was appointed manager.

Robert J. Rose has gone to the Union of S. Africa where he can be reached in care of the Messina Development Co., Messina, Transvaal.

Pierre Routhier, fellow of the University of Paris, former head of the geological mission of the office of the Recherche Scientifique Coloniale in New Caledonia, left New Caledonia recently. He and two associates will study the material collected there in the geological laboratory of the Ecole Normale Superieure, 45 rue d'Ulm, Paris.

Larry Seaman, project manager for the Iron Mines Co. of Venezuela, a Bethlehem Steel Co. subsidiary, has changed his address from Caracas, Venezuela, to Guiria, Estado Sucre, Venezuela, where the Iron Mines Co. is building a port for the transshipping of Venezuelan iron ores.

Henry G. Schuring, formerly connected with La Luz Mines Ltd. at Siuna, Nicaragua, is now mill superintendent of the New Idria Honduras Mining Co., Santa Rosa de Copan, Honduras.

Charles E. Stott, formerly vice-president and general manager of Cia. Minera de Penoles and Cia. Metalurgica Penoles, subsidiaries of the American Metal Co., is at present located in Washington, D. C., as regional specialist with the strategic materials division of ECA, covering Africa.

J. A. Swart, who was general superintendent for the Raymond Concrete Pile Co., on their contract to build the Cardon refinery for the Shell Oil Co., has gone to Chuquicamata, Chile, as assistant job manager with Foley Hermanos on the construction contract to build a new sulphide plant for the Chile Exploration Co.

J. R. Sweet arrived in the States last August from Northern Rhodesia where for the past 4½ years he was in the service of the Roan Antelope Copper Mines, Ltd., Luanshya. He is now general manager for the Tungsten Mining Corp., Henderson, N. C. His residence address is 230 Virginia Ave., Henderson.

E. H. Thaete, Jr., has transferred his headquarters from New York to the New Orleans office of the Freeport Sulphur Co., 1804 American Bank Bldg.

E. L. Thomas, Jr., was graduated in mining engineering from the University of Alabama at the end of the last quarter and will be located in the Birmingham district with the Tennessee Coal, Iron, and Railroad Co.

David W. Tittman graduated from the Missouri School of Mines in January and was then employed by the St. Joseph Lead Co. as metallurgist in the ore dressing research laboratory. He is living at 207 E. School St., Bonne Terre, Mo.

Hale C. Tognoni is working at the Darwin Mines, Darwin, Calif. He had been a student at the University of Nevada.

Marvin J. Udy now spends half of his time with the Vanadium Corp. of America in the development of new processes for chromium and ferroalloys. At the same time he is acting as consulting engineer for the New Jersey Zinc Co. in the electric furnace smelting of Quebec ilmenite, and also for Fabrica Nacional de Carburo y Metallurgia of Santiago, Chile.

Gilfry Ward has been appointed vice-president in charge of sales of the American Manganese Steel Division of the American Brake Shoe Co. He will be located in Chicago Heights, Ill. With Brake Shoe since graduating from Yale in 1928 he has spent many years on both the East and West Coasts with the Amsco sales organization.

H. P. Willard has changed his address from 166 Tomb St., Tiffin, Ohio, to Gabbs, Nev.

J. P. Williams, Jr., chairman of the board of directors of the Koppers Co., Pittsburgh, retired from active management of the company in May. He remains a member of the board and will continue in his capacity as chairman of the board, as well as an adviser on special problems. Actively associated with the Koppers Co., or its predecessors, since 1920, he was manager of the Melcroft Coal Co. until Koppers absorbed it, was elected executive vice-president of Koppers in 1933, and became president in 1939.

John Worcester has been appointed production manager of the National Lead Co., S. A., with headquarters in Buenos Aires, Argentina.

Carl C. Youell is superintendent of the Jewell Ridge Coal Corp. at Oneida, Tenn.

• In the Metals Branch

© Harris and Ewing

Robert L. Baldwin

Robert L. Baldwin has been made assistant to **G. A. Wallerstedt,** western district manager for the Hardinge Co., York, Pa. He will assist in sales development, using the Hardinge office at 24 California St. in San Francisco as headquarters. Mr. Baldwin began his career in 1901 as a technician for the American Steel and Wire Co. in Cleveland. He joined the U. S. Steel Corp. in 1912 as a special representative in exploiting, licensing, and selling the Heroult electric steel furnace. When the Heroult basic patent expired in 1920 he joined the Republic Carbon Co., which was acquired fourteen years later by the National Carbon Co. He became sales development manager of the electrode division of that organization, retiring from that post this year to join the Hardinge organization.

Thomas E. Barlow, formerly on the staff of Battelle Memorial Institute, is now sales manager for Eastern Clay Products, Inc., Jackson, Ohio.

Joseph J. Brugman is no longer employed by Idaho Smelting Inc. He is now with the Morley Magnesium Foundries, Box 628, Renton, Wash., as metallurgical superintendent.

Thornton C. Bunch has transferred to Vallejo, Calif., where he is foundry superintendent at the Mare Island Naval Shipyard. He had been working at the Navy Yard at Pearl Harbor.

J. E. Burke, formerly at the University of Chicago, has recently joined the Knolls Atomic Power Laboratory at the General Electric Co. in Schenectady as a research assistant. His new home address is R. D. 1, Ballston Lake, N. Y.

Hans I. Elvander, Box 3616, Degerfors, Sweden, is assistant to the general superintendent of Degerfors Jernverks AB.

Matthew A. Hunter is retiring as dean of faculty after forty years of service at Rensselaer Polytechnic Institute, Troy, N. Y. He joined the faculty in 1908 as a professor in electrochemistry and has served as the head of the departments of electrical engineering, physics, and metallurgical engineering. Dr. Hunter was born in Auckland, New Zealand, in 1878, received a B.S. and M.A. degree from Auckland University College and then went on to London for a doctor of science degree from University College. He continued his studies at the University of Gottingen, Germany, at the Sorbonne in Paris, and the Techniche Hochschule at Karlsruhe, and came to the States in 1905 to work in the General Electric research laboratory in Schenectady.

Thomas A. Jackson is engaged as

a metallurgist and representative for the Volcan Mines Co. at Mahr Tunnel, Peru.

Edward C. Keller, formerly plant metallurgist with the Duquesne Smelting Corp. at Pittsburgh, has joined the firm of K. Hettleman and Sons at Baltimore, Md., as assistant manager of the Dixie metals division.

George V. Luerssen, chief metallurgist for the Carpenter Steel Co., was presented with the first annual David Ford McFarland award by the Penn State Chapter of the ASM at a dinner meeting on May 12. This award is made annually to a Penn State metallurgist who is judged to have attained eminence in his profession so as to bring credit upon himself and his alma mater. Mr. Luerssen talked on iron-nickel alloys and emphasized the need for further metallurgical attention in this field.

Robert E. Mahr has resigned his job with Sylvania Electric Products Inc. and is now located with the Chrysler Corp., central engineering division, powdered metals lab. in Highland Park, Mich. He will be glad to hear from any friends at his home at 2360 Bacon Ave., Berkley, Mich., phone number Lincoln 2-8728.

Daniel J. Milevich is mechanical foreman in the open hearth department of the International Harvester Company's Wisconsin Steel Works at S. Chicago, Ill.

Martin S. Miller is research metallurgist in the research laboratory of the Crucible Steel Co. of America at Harrison, N. J.

Herbert E. Pagel is now in Birmingham, Ala., working for the Tennessee Coal, Iron and Railroad Co. as a laboratory assistant in the metallurgical department in the Fairfield sheet mill.

Clarence B. Randall was elected to the presidency of the Inland Steel Co. on April 27 to succeed **Wilfred Sykes,** who becomes chairman of the executive committee. Mr. Randall, who had been assistant to the president, was vice-president in charge of materials. Mr. Sykes advised stockholders that net income for the quarter ended March 31 amounted to $9,254,230 or $1.89 a share, compared with a net of $8,458,544 or $1.73 for the March quarter last year. Mr. Sykes reported that there is now "ample evidence" that the pent-up demand for steel following the war is being satisfied, and that Inland's and the industry's capacity will be more than sufficient to supply the needs of the country, even at maximum peacetime consumption.

L. F. Reinartz has been named assistant vice-president of the Armco Steel Corp. He will continue to manage the Middletown division of Armco and the company's coal mining division in West Virginia will report to him. Mr. Reinartz began his Armco career in 1909 as a chemist in the open hearth department after receiving a metallurgical degree from Carnegie Institute of Technology. He advanced from assistant superintendent to superintendent of the open hearth department at Middletown, to assistant general superintendent to works manager of East works and in 1938 to manager of the Middletown division.

F. W. Bremmer

F. W. Bremmer, for 25 years with the National Supply Co., has been elected vice-president in charge of manufacturing, a post which has been vacant since **Charles R. Barton** retired a year ago. Mr. Bremmer has been works manager of the Spang-Chalfant division's Ambridge plant since May, 1948. He was first employed by the Standard Seamless Tube Co. of Ambridge, Pa., and shortly after that company was acquired by Spang-Chalfant became assistant superintendent of the hot mills. Spang Chalfant was acquired by National Supply in 1930 and ten years later Mr. Bremmer was appointed works manager of the division's Etna, Pa., plant, a post he held until his appointment as works manager of the Ambridge plant last year.

Earl W. Ross, who had been with the Naval Shipyard in Brooklyn, N. Y., has gone to Teterboro, N. J., to work for the Bendix Aviation Corp.

Bruce M. Shields has changed his address from the U. S. Steel Corp. of Delaware, Pittsburgh, to the metallurgy department of the Carnegie-Illinois Steel Corp. South Works, 3426 E. 89th St., Chicago 17.

Eric G. Skarin is service metallurgist for the Ohio Ferro-Alloys Corp., Citizens Bldg., Canton 2, Ohio.

Cyril Stanley Smith, director of the Institute of Metals at the University of Chicago, was one of the principal speakers at the dedication of two new buildings on the campus of Illinois Institute of Technology on June 17. He lectured and led a discussion on the "Microstructure of Metals" at a symposium in the auditorium of the chemistry building, one of the two being dedicated. The other new structure is the metallurgical and chemical engineering building.

F. M. Stearn, formerly with the Appleby Frodingham Steel Co., is now assistant metallurgist in the process development department of Henry Wiggin & Co., Birmingham, England.

Ralph H. Sweetser writes from his new address, RFD 2, Portsmouth, N. H., that he spent four months in Ohio in the midst of the controversy on the "rubber conveyor belt system" from Lorain to East Liverpool, Ohio, for conveying iron ore from the Lake to the Ohio River and coal from the River to the Lake. Mr. Sweetser fails to see how this will help the steel industry and feels that it won't help the railroads. He has found quite an interest in sponge iron, but doesn't know whether it is because of the scarcity and high cost of blowing engines or because of the tests of the Bureau of Mines at Laramie, Wyo.

Elbert C. Troy, who was chief metallurgist for the Dodge Steel Co., is now foundry engineer for the National Engineering Co. He receives mail at 5 E. Broad St., Palmyra, N. J.

J. C. Warner, dean of graduate studies and head of the chemistry department at Carnegie Institute of Technology, has been elected vice-president and a member of the board of directors of the Electrochemical Society.

D. F. Zlatnik is plant superintendent for the Basic Reduction Co. on the hydrometallurgical extraction, recovery, and refining of lead and zinc. His mail goes to P. O. Box 92, Henderson, Nev.

• In Petroleum Circles

John A. Fraher, formerly with the Sinclair Prairie Oil Co., is now employed as a petroleum engineer by the Bureau of Mines, Box 621, Laramie, Wyo. He is working on secondary recovery problems in the Rocky Mountain era.

Earl G. Griffith, after a little over three years with the Mene Grande Oil Co. in the Maracaibo Lake basin, the last year and a half as resident geologist of the Bolivar Coastal fields, decided that it was time to put in a "contract" in the U. S. Moreover he and his wife wanted their son to be born in the States and enjoy the advantages of good medical care. Mr. Griffith arrived home 24 hours prior to his son's birth, which was pretty close timing. At present he is a petroleum geologist with The Texas Co. in the Montana district. He is living in Lewistown, Mont., and thinks it good country, with lots of hunting and fishing.

James W. Hepburn recently received appointment as assistant manager of the vertical turbine pump division of the Worthington Pump and Machinery Corp., Harrison, N. J. Mr. Hepburn set up his headquarters at the Corporation's Denver Works early in May, where he will be responsible for customer relations in the sale of vertical turbine pumps in the territories west of the Mississippi, including Chicago, St. Paul, and New Orleans. Mr. Hepburn was graduated from the engineering school of the University of Utah in 1929 and joined Worthington that same year as a sales engineer. In 1943 he was made district manager of the El Paso office, and since 1945 has been in the reciprocating pump division at the Harrison Works.

Michel J. Pagezy, a French student who had been studying at the Missouri School of Mines, has been in training as a petroleum engineer in production with the Sinclair Wyoming Oil Co. He expected to return to France in July to offer his professional services to the French petroleum industry.

Robert H. Parks has moved to San Antonio, Texas, and represents the Sunray Oil Corp. on the Seeligson Engineering Committee. His address is Transit Tower, San Antonio.

C. Sheldon Sharp has gone to Dhahran, Saudi Arabia, where he will be employed as a geophysicist working with gravity exploration for the Arabian American Oil Co. For the past ten years he had been working for the Brown Geophysical Co. on gravity surveys.

J. H. Sidwell has changed his address from the Union Tank and Supply Co., Tulsa, to the Hiawatha Oil and Gas Co., Milan Bldg., San Antonio, Texas.

M. H. Stekoll informs us that the main office of the Stekoll Petroleum Co. is now located at 1700 M&W Tower Bldg., Dallas. He has recently moved from Independence, Kan., where the Company, which is engaged in the oil producing business, has several waterflood operations.

Munsey W. Waters, engineer for the George E. Failing Co., manufacturer of rotary drilling equipment, can be reached at 1001 Sunset Drive, Enid, Okla.

Bing Quai Yee is addressed at the exploration department of the Carter Oil Co., P. O. Box 120, Denver, Colo. He had been at Oklahoma University.

Robert W. Zwicky has the job of junior exploitation engineer with the Shell Oil Co. and receives his mail at 2436 Shakespeare, Houston, Texas.

Obituaries

CHASE SALMON OSBORN (Member 1916), former governor of Michigan, prospector, geologist, writer, and a man who helped create some of the history of his times, died in Poulan, Ga., on April 11, at the age of 89.

Mr. Osborn's Lincolnesque, self-made career began in an Indiana log cabin in 1860, and his first job was that of newspaper boy. By 1912 he had bought, published, and sold three mid-western newspapers. A penchant for hunting took him off on expeditions to the Canadian woods, and led to his discovery of the Michipicoten iron range and the Moose Mountain range in that country's northland, which discoveries brought him a considerable fortune. He was instrumental in the development of the Great Lakes region, and did much field work in the Lake Superior districts of Michigan, Wisconsin, Minnesota, and Ontario.

The efforts of "Uncle Chase" were not confined to the United States, however, as witness his discovery of the Kiruna and Luossavara deposits in Lapland, and other iron ranges in Africa, the Orient, and Latin America. His adventures in Madagascar were presented in his book "Madagascar, Land of the Man-Eating Tree," in 1924.

Mr. Osborn held no less than seven university degrees, although he had only that number of years of formal schooling. The degrees were honorary—honoring the man who was Michigan's governor from 1911 to 1913; who wrote one of the nation's first workmen's compensation laws; who was instrumental in drafting Theodore Roosevelt for the presidency; who founded the Michigan Fellowship in Creative Art; who headed the National Committee of Independent Voters for Roosevelt in the 1940 campaign; who knew nine Presidents, and gave away a fortune.

The list of organizations of which he was a member would fill most of this column. As an author, he wrote "The Andean Land"; "The Iron Hunter"; "The Law of Divine Concord"; "A Short History of Michigan"; and other volumes.

Perhaps he left a clue to the secret of his success. Someone once asked him what schools he had graduated from, to which he replied—"Big woods and mountains."

ANDREW MELLICK TWEEDY (Member 1914), mining engineer, and former director and general manager of the South American Development Co., died at San Angelo, Texas, on Jan. 6. He was 64 years old. Mr. Tweedy was a native Texan, but he came East to take his B.S. in mining engineering from the Columbia School of Mines in 1906. He worked for the American Pyrites Co. in Herman, N. Y., for two years, and then the next eight years found him employed in various Mexican mining camps. In 1916, he became resident manager for the South American Development Co. in Portovelo, Ecuador. Thirteen years later he was managing the company from its New York offices, and in 1935 became a director, continuing in that post until his retirement in 1943. His services were retained as a consulting engineer for the firm until the time of his death.

One of his noteworthy achievements was the placing of the old Zaruma operation on a profitable basis, and Mr. Tweedy's ability, vision, and understanding were well known to have played a major part in the solution of the many problems therein presented.

Necrology

Date Elected	*Name*	*Date of Death*
1948	Robert H. Bedford	April 26, 1949
1948	Charles A. Bruce	March 11, 1949
1892	William H. Corbould	March 16, 1949
1948	William J. Flori	May 7, 1949
1896	Robert H. Jeffrey	January 31, 1949
1920	Joseph P. Labaw	April 27, 1949
1909	James E. Little	May 24, 1949
1947	Leonard Lynch	Oct. 18, 1948
1890	James MacNaughton	May 26, 1949
1909	Charles Mentzel	June 1, 1949
1916	Hubert Merryweather	June 7, 1949
1912	William C. Phalen	May 27, 1949
1945	Russell E. Reilly	Unknown
1917	Chard O. Sanford	January 31, 1949
1945	George L. Smith	Unknown
1918	Philip S. Smith	May 10, 1949
1915	Walter Stalder	May 27, 1949
1938	Arthur B. Yates	May 10, 1949

Proposed for Membership

Total AIME membership on May 31, 1949, was 15,729; in addition 4348 Student Associates were enrolled.

In the following list C/S means change of status: R, reinstatement; M, Member; J, Junior Member; AM, Associate Member; S, Student Associate; F, Junior Foreign Affiliate.

ARIZONA

Ajo—NEWETT, JOHN W. (M). Chief engineer, New Cornelia branch, Phelps Dodge Corp.

Globe—MELLUS, HERBERT HEATH. (C/S—J-M). Pit engineer, Inspiration Consolidated Copper Co.

Inspiration—PATE, MANOAH LASLEY. (C/S—J-M). Mining engineer, Inspiration Consolidated Copper Co.

Tucson—KUMKE, CHARLES AUGUST. (R-M). Mining engineer, Bureau of Mines.

ARKANSAS

Smackover—VESTAL, WILLIAM ARTHUR. (C/S—S-J). Petroleum engineer, Phillips Petroleum Co.

CALIFORNIA

Hollywood—OFFEMAN, RICHARD E. (M). Manager, Pacific Coast division laboratory, Baroid Sales Division, National Lead Co.

Los Angeles—HAYTHORNE, PERCY ALLEN. (M). Research engineer, supervisor of metallurgical research, Lockheed Aircraft Corp.

Riverside—NICKERSON, KENNETH WESLEY, JR. (C/S—S-J). Geologist, Kennecott Copper Corp.

Rodeo—MONFORT, JOSE VARGAS. (C/S—J-M). Smelter chemist, American Smelting and Refining Co.

San Carlos—HACK, WALTER LAWRENCE. (M). Chief metallurgist, Western Gold and Platinum Works.

Taft—HENDERSON, ALEXANDER DUNCAN, JR. (R,C/S—JA-M). Field superintendent, Stanolind Oil Co. of California.

COLORADO

Denver—UNDERWOOD, ERVIN EDGAR. (C/S—J-M). Graduate student, MIT.

Leadville—SCHULTZE, JOHN FREDERICK. (C/S—S-J). Metallurgist, Leadville milling unit, American Smelting and Refining Co. YOUTZ, ROBERT CHARLES. (C/S—S-J). Mine sampler, Resurrection Mining Co.

ILLINOIS

Chicago—FERM, ERIC W. (M). Sales metallurgist, Miller & Co. MARTIN, HAROLD JOHN. (M). Sales engineer, Miller & Co.

Du Quoin—ALBON, DEAN ELLIOTT. (J). Chief engineer, Airmite-Midwest, Inc.

Wilmette—WARE, THOMAS MORROW. (M). Industrial engineer, International Minerals and Chemical Corp.

INDIANA

E. Chicago—BROWN, CALVIN CHARLES. (C/S—J-M). Metallurgist, Inland Steel Co. TATOUSEK, RICHARD JOSEPH. (C/S—J-M). Open hearth practice foreman, Inland Steel Co.

Fort Wayne—LAULETTA, PAUL ANDREW. (C/S—J-M). Metallurgical engineer, Joslyn Mfg. & Supply Co.

Hammond—BONICK, JAMES FRANCIS. (J). Metallurgist, Inland Steel Co.

LOUISIANA

New Orleans—BANKS, ROBERT TUNSTALL, JR. (M). Consulting engineer. GLENN, ALFRED HILL. (M). Consulting meteorologist, A. H. Glenn & Associates. SQUYRES, FRANK WILBUR. (AM). District representative, Eastman Oil Well Survey Co.

Shreveport—FENWICK, DICK TIMS. (M). Production supt., Southern Production Co. REEVES, JAMES NEAL. (J). Geological scout, Pan American Production Co.

MARYLAND

Silver Spring—BACON, JAMES EVERETT. (M). Technical counselor.

University Park—BARRY, ANTHONY J. (M). Mining engineer, Research, Bureau of Mines.

MASSACHUSETTS

Cambridge—GAINES, RICHARD VENABLE. (C/S—J-M). Postgraduate student, Harvard University. JAFFE, LEONARD DAVID. (C/S—J-M). Chief, physical metallurgy section, Watertown Arsenal. MEAD, JUDSON. (C/S—S-M). Geophysicist, U. S. Smelting Refining & Mining Co.

Newton—MARTIN, JAMES LEO. (C/S—J-M). Director-laboratory, Watertown Arsenal. PITTS, PARKER DESALES. (J). Geologist, F. D. Pitts Co.

Springfield—MORRIS, ALBERT WOOD. (R-M). Research engineer, director of research, Moore Drop Forging Co.

Wellesley Hills—STOKES, CHARLES ANDERSON. (M). Director of research and development, Godfrey L. Cabot, Inc.

MICHIGAN

Ann Arbor—REYNOLDS, EDWARD EVANS. (J). Research associate, Engineering Research Institute, University of Michigan.

Ishpeming—RIBOTTO, PETER PAUL. (C/S—J-M). Chief engineer, Inland Steel Co.

MINNESOTA

Buhl—SCHEUER, CHARLES HENRY. (M). Chief engineer, W. S. Moore Co.

MISSISSIPPI

Washington—DONALDSON, ROBERT WARREN. (J). Petroleum engineer, The California Co.

MISSOURI

Kirkwood—MOORE, THOMAS I. (C/S—J-M). Assistant general superintendent, American Zinc Co. of Illinois.

Rolla—DAVIS, CHARLES MALCOLM. (M). Assistant professor of petroleum engineering, mining department, Missouri School of Mines.

St. Louis—BURKHARDT, FRED ROLLAND. (M). Civil engineer, Sinclair Coal Co.

MONTANA

East Helena—LANE, STANLEY MARTIN. (R,C/S—J-M). Assistant superintendent, American Smelting & Refining Co.

NEVADA

McDermitt—HAAS, VERNE P. (M). General superintendent, Cordero Mining Co.

Pioche—McINTYRE, E. STEELE. (C/S—J-M). Mine engineer, Combined Metals Reduction Co.

NEW JERSEY

Freehold—REICHARD, EDMUND CARL. (C/S—J-M). Research metallurgist, American Smelting & Refining Co.

Union—WALLACE, EDWARD MORRIS. (C/S—J-M). Chief engineer, Vulcan Safety Razor Corp.

NEW MEXICO

Hanover—CASON, CARROLL C. (C/S—S-J). Engineer, Peru Mining Co.

Hobbs—BRIGHT, WAYNE R. (R,C/S—S-M). Petroleum engineer, Stanolind Oil & Gas Co. COURTRIGHT, RAYMOND ORLAND, JR. (M). Field engineer, Stanolind Oil & Gas Co.

NEW YORK

Floral Park—BEYER, ARTHUR JONES. (C/S—J-M). Metallurgical engineer, Atomic Energy Commission.

New York—KAHN, WALTER CHARLES, JR. (C/S—J-M). Assistant technical director, N. Y. Testing Laboratories, Inc. REED, HARLOW JOHN. (C/S—J-M). Metallurgical engineer, Singmaster & Breyer. SCARBOROUGH, FRANK GRIFFIN. (C/S—S-J). Engineer officer, fleet training group, U. S. Navy, Guantanamo Bay. WANG, KUNG-PING. (R,C/S—S-M). Specialist, foreign minerals, Bureau of Mines. WAYLAND, RUSSELL G. (C/S—J-M). Special adviser, combined coal control group, OMGUS.

Schenectady—GEISLER, ALFRED HOLLIS. (C/S—J-M). Research associate, General Electric Co.

NORTH CAROLINA

Charlotte—CAMPBELL, JAMES EDWARD. (C/S—J-M). Chief smoke abatement engineer, City of Charlotte, N. C.

Raleigh—BROADHURST, SAMUEL DAVIS. (AM). Assistant state geologist, State of North Carolina.

OHIO

Cleveland—LYMAN, TAYLOR. (C/S—J-M). Associate editor, *Metal Progress* and editor, *Metals Handbook*, ASM.

Columbus—RABB, DAVID DANIEL. (C/S—J-M). Research engineer, Battelle Memorial Institute.

Youngstown—SPEER, EDGAR BOYLE. (M). Superintendent, Bessemer department, Carnegie-Illinois Steel Corp.

OKLAHOMA

Enid—WATERS, MUNSEY W. (C/S—AM-M). Engineer, George E. Failing Supply Co.

Tulsa—STINSON, JAMES ALBERT. (M). Petroleum engineer, Stanolind Oil & Gas Co.

PENNSYLVANIA

Aliquippa—KELLER, ALONZO CARROLL. (M). Superintendent, open hearth & Bessemer, Aliquippa works, Jones & Laughlin Steel Corp.

Butler—PARKER, HAROLD MERRITT. (M). Open hearth superintendent, Armco Steel Corp.

Latrobe—FLETCHER, STEWART G. (C/S—J-M). Chief metallurgist, Latrobe Electric Steel Co.

New Kensington—PHILLIPS, GLEN L. (C/S—J-M). Operating engineer, Aluminum Co. of America.

Philadelphia—FAY, MERVIN A. (C/S—J-M). Ceramic engineer, General Refractories Co.

Pittsburgh—BROWN, BENJAMIN FLOYD. (C/S—J-M). Research assistant, metals research laboratory, Carnegie Institute of Technology. DILLON, HERBERT GEORGE. (M). Sales manager, Heyl & Patterson, Inc. ERKUN, NEVZAT MEHMET. (C/S—S-J). Metallurgist, Pittsburgh Forgings Co. PETTIGREW, DAVID WEBB, JR. (C/S—S-J). Research engineer, research laboratory, Aluminum Co. of America.

Plymouth—BIRD, MORGAN, JR. (J). General manager, Kingston Mining Corp.

Riegelsville—ADAMS, JOHN H. (C/S—S-AM). Geologic field assistant, U. S. Geological Survey.

Rimersburg—WALKER, JOSEPH LAWRENCE, JR. (C/S—S-J). Resident mining engineer, Rimersburg Coal Co.

State College—LECZNAR, FRANCIS JOSEPH. (M). Assistant professor, Academy of Mining in Krakow.

TENNESSEE

Knoxville—BERGMAN, GEORGE WALTER. (M). District manager, Joy Mfg. Co.

TEXAS

Conroe—HAINES, FRANK SAMUEL, JR. (C/S—S-J). Field engineer, The Texas Co.

Corpus Christi—MONTGOMERY, JAMES M. (M). Tools superintendent, Halliburton Oil Well Cementing Co.

Dallas—WISENBAKER, JOHN D. (M). Manager of operations, Core Laoratories, Inc.

Denver City—CAWLEY, AARON. (C/S—S-J). Engineering trainee, Honolulu Oil Corp.

El Paso—CHRISTENSEN, FRANK J. (M). Construction engineer, district manager, Stearns-Roger Mfg. Co.

Graham—WESTFALL, MILLARD FRANK. (J). District petroleum engineer, Cities Service Oil Co.

Houston—JONES, KENNETH E. (M). Division engineer, Baash-Ross Tool Co. WEAVER, EARL MARSHALL. (M). Vice-president, Herb J. Hawthorne, Inc. WESTBROOK, M. ALLEN. (M). Supervisor, Houston area production laboratory, Shell Oil Co.

Lubbock—ENGLE, DONALD DAVID. (J). Junior petroleum engineer, Stanolind Oil & Gas Co. HINKLE, HENRY WILLIAM. (J). Equipment engineer, Stanolind Oil & Gas Co. THOMPSON, LEWIS MARTIN. (J). Petroleum engineer, Stanolind Oil & Gas Co.

McAllen — McGEE, CHARLES J. (C/S—S-J). Engineer and branch office manager, Petroleum Service Co.

Midland — FORGERSON, CARL DAVIS. (J). Junior petroleum engineer, Stanolind Oil & Gas Co. MONTGOMERY, WILLIAM JEFFERSON. (C/S—S-J). Lawyer, Klapproth & Hamilton. WRIGHT, THOMAS RUSSELL. (R,C/S—S-J). Junior petroleum engineer, Stanolind Oil & Gas Co.

North Cowden — WILLIAMSON, JACK WOODROW. (M). Petroleum engineer, Continental Oil Co.

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Tyler — DeLONG, JACK MYLER. (AM). Geologist, Stanolind Oil & Gas Co.

Yorktown — SUTHERLAND, JOHN WELSEY, JR. (R,C/S—S-J). District engineer Sunray Oil Corp.

UTAH

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INDIA

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VICTORIA

Melbourne—CLARK, GORDON COLVIN LINDESAY. (M). Technical managing director, Western Mining Corp., Ltd.

BORNEO

Sarawak—ROE, FRANK WALTON. (M). Chief geologist, British Territories in Borneo.

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Controlled Drying of Retorts

R. R. FURLONG* and D. H. WERTZ*

Dry room equipment at Donora Zinc Works is of the design which prevailed at the time the plant was built in 1915. It consists of 11 adjoining rooms, each being 99 ft long, 11 ft wide, and 7 ft high and having a capacity for 1040 retorts. Heat is supplied by steam coils beneath the slatted wood floor and by warm air ducts at the ceiling. Drying practice is about the same as that generally followed in the industry except that, under normal zinc furnace operations, the maximum drying time is 45 days.

During certain seasons of the year the drying process was seriously affected by varying weather conditions which frequently resulted in a scrap loss of 5 to 7 pct. Because of these unfavorable conditions, which caused uneven drying and strains in the retorts, increased retort failures in the zinc furnaces were recorded. Regulation of temperature and humidity under all conditions appeared to be the logical solution of this problem.

Controlled drying had been developed for a number of ceramic products and investigation of several such drying processes in this area revealed favorable results. Along with control of drying conditions and the attendant decrease in scrap losses, plant operators also reported a material reduction in drying time.

Another factor affecting the decision to proceed with this development was the drastic change in market conditions for retort clay. Curtailment of shipments from the chief source of supply made it necessary to proceed with blending of other available clays, but quality of these clays was questionable for adequate retort life in the 24 hr furnace cycle. Rapid furnace testing of new mixtures was desirable but impossible with the prolonged drying schedule. Therefore development of a controlled drying process for zinc retorts offered the solution to the problem of rapid testing of retort mixtures.

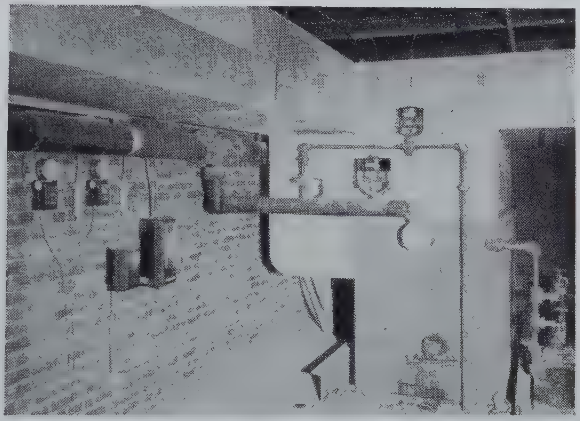

FIG 1—Front view of Carrier Heater unit.

Description of Process and Equipment

The freshly extruded retort is cylindrical in shape, 58 in. long, 9 in. id and has 1⅛ in. side walls. One end is closed and approximately 2 in. thick. The moisture content of this "green" retort is about 17 pct which means a necessary removal of 30 lb of water per retort. Retorts are set on the slatted floor of the dry room with the closed end down.

The controlled drying process depends upon the circulation of a large volume of conditioned air at low velocity. To provide for uniform distribu-

San Francisco Meeting, February 1949.

TP 2555 D. Discussion of this paper (2 copies) may be sent to *Transactions* AIME before Aug. 15, 1949. Manuscript received October 28, 1948.

* Foreman, Pottery Department, and Ceramic Engineer, respectively, American Steel and Wire Co., Donora Zinc Works, Donora, Pennsylvania.

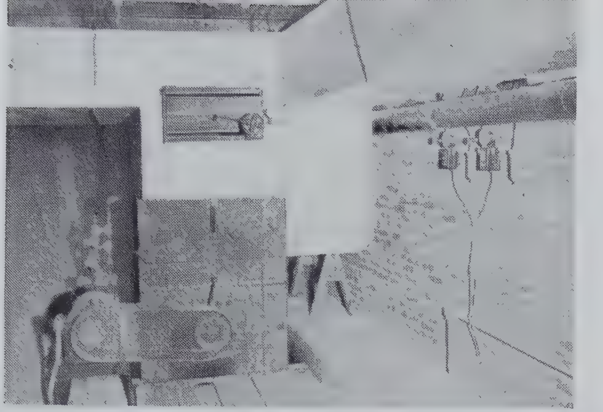

FIG 2—Rear view of Carrier Heater unit.

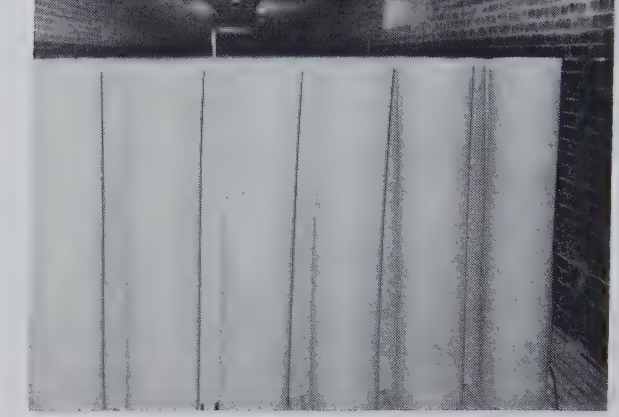

FIG 3—View of retorts in drying room.

tion of the air stream, the heater units should discharge into a large chamber. The only available space for this, in the existing dry rooms, was under the slatted floor. Two inverted type Carrier Heater units, each with a fan capacity of 17000 cfm, were installed in the middle one of three dry rooms, and connected to each adjacent room with short ducts leading into wall openings above and below floor level. Manually operated interlocked dampers within the unit regulate the flow of air to either room.

To control conditioning of the air stream, each heater unit is provided with wet and dry bulb temperature instruments located beneath the floor of each room opposite the duct opening. These control instruments actuate air operated valves on the steam lines to the heater coils and to the steam spray (Fig 1). These supply heat and moisture as required. During the drying cycle a slight pressure is maintained in the dry room and make-up air is introduced through a fresh air damper (Fig 2). This damper, located in the rear wall of the heater unit above the steam coils, is operated by an air valve which in turn is controlled by the dry bulb instrument.

After considerable experimental work a standard practice for quick controlled drying of zinc retorts has been developed to suit conditions at this plant.

Loading of the room requires about two days production from the retort press. Retorts are placed in staggered rows 3 in. apart and this widespacing provides free movement of the rising warm air around the retorts (Fig 3). Incidentally, this spacing has reduced the capacity of each room from 1040 to 760 retorts.

After loading, the stack damper and doors of the room are closed and retorts are allowed to stand at existing conditions until the following morning. Air circulation is then started and continued for 48 hr at a dry bulb temperature of 120°F and 72 pct relative humidity. The wet bulb temperature for this humidity is 110°F which temperature remains constant throughout the drying cycle. On the third day the dry bulb temperature is held at 130°F for 8 hr with a relative humidity of 53 pct. For the next 16 hr the dry bulb temperature is fixed at 140°F with a corresponding decline in relative humidity to 38 pct. During the first 6 hr of the fourth day the dry bulb temperature is increased in 10° intervals until 170°F is reached, with a final relative humidity of 17 pct. At this point the fresh air and stack dampers are opened. Fresh air heated to 170°F is allowed to sweep through the room for the next 16 hr to complete the drying cycle. Steam is then shut off and the fan is used to cool the room.

Summary

1. It is evident from the above description of drying practice that the principles of controlled drying can be beneficially applied to the manufacture and quick testing of zinc retorts.

2. The drying cycle has been reduced from 45 days to 4 days with assurance that drying conditions can be duplicated. This feature is extremely valuable in the rapid testing of new retort mixtures.

3. Retort losses in drying have been reduced to less than one percent. This is about half the normal loss expected with the slow drying practice.

4. Close regulation of temperature and humidity has eliminated their fluctuation caused by variations in the weather.

5. Numerous zinc furnace tests have not shown any failure of retorts which could be associated with the quick drying process. At the same time no improvement in service in the furnaces has been detected.

6. The capacity of the present installation is not large enough to supply total retort requirements for current furnace operations. Therefore the equipment is used to quick-test the quality of retort mixtures to be dried by the slow process, and as additional capacity for emergency production. In these respects the installation has been satisfactory.

7. Quick drying equipment of sufficient capacity for the entire smelting furnace requirements would reduce the number of drying rooms by one-half with a corresponding decrease in retort inventory.

The Study of Grain Boundaries with the Electron Microscope*

J. F. RADAVICH†

Introduction

Many heats of steel of low carbon value have been known to produce brittle pieces of steel. The brittleness is believed to be due to the impurities located within the grain boundaries. Such brittle steels have been examined with an optical microscope to ascertain the nature and the amount of the impurities present at the grain boundaries. Due to the relatively low resolving power of the optical microscope, the impurities are not visible in fine detail.

The writer obtained some sheet steel and proceeded to determine the location of the impurities and to show the application of the electron microscope to the study of grain boundaries. One sample was known to be capable of becoming embrittled, whereas another sample was believed to be much less susceptible to embrittlement.

Treatment of Specimens

The specimens were embrittled by annealing above the A_3 point under mildly oxidizing conditions. One piece of ingot iron could not withstand a 90° bend, whereas another piece of ingot iron was not affected and could withstand a 90° bend. The brittle piece was then annealed at a high temperature in a hydrogen atmosphere.

The annealed ingot iron was termed *cured* and could withstand a 90° bend very easily. The three specimens examined will be designated as *brittle*, *good*, and *cured* in the discussion that follows.

Procedure

The sizes of the specimens were as follows: one piece of brittle ingot iron–⅜ by ⅛ in.; one piece of good ingot iron–⅜ by ⅛ in.; one piece of cured ingot iron–⅜ by ⅛ in.

The specimens were too small to be polished by hand and therefore were mounted in bakelite. The polishing procedure was carried out in the conventional manner with the use of 1/0 through 3/0 papers, and the final polish was done with alumina on a billiard cloth. The specimens were then etched in a 4 pct solution of picral in alcohol, and then they were examined through an optical microscope. An area was chosen that showed distinct grain boundaries, and an effort was made to keep near this area when pulling the replicas.

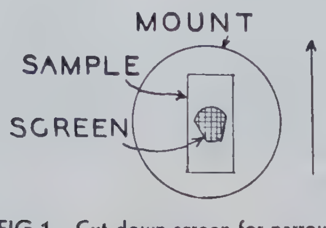

FIG 1—Cut-down screen for narrow specimens.

REPLICA TECHNIQUE

The replica technique used in the preparation of the replicas for examination under the electron microscope is described in Electron Metallography.[1] It consists essentially of the following steps:

1. Obtaining a suitably etched specimen.
2. Applying a swab of ethylene dichloride on the surface.
3. Applying a formvar solution on the surface.
4. Placing a screen on any desired spot.
5. Breathing on the formvar layer.
6. Applying scotch tape on the screen and film.
7. Pulling the film and the screen up with the Scotch tape.
8. Separating the screen from the Scotch tape.

This replica technique is very similar to the one described by Harker and Shaefer.[2] However, with the added step, the percentage of replicas removed is very much higher regardless of the length of the time from the etching of the specimen to the actual pulling of the replica.

The replicas were then shadow cast with manganese at a filament height to replica distance ratio of 1½ :7. This produced a very high contrast replica for use in the electron microscope.

One of the difficulties encountered with this study was the restricted area of the specimen. The width of the specimens was the same as that of the 200 mesh nickel supporting screen. In order to increase the effective area, the screens were cut down as shown in Fig 1. The arrow indicates the direction in which the replica was pulled. This operation made it possible to obtain a large percentage of good replicas.

Fig 3 shows an electron micrograph of a brittle piece of ingot iron and a grain boundary that was polished mechanically. The surface is very rough probably due to the incomplete removal of the flowed layer by the picral etchant. The grain boundary does show evidence of impurities. It was decided to electropolish the specimens to obtain a much smoother surface than the one obtained by mechanical polishing.

ELECTROPOLISHING

The specimens were cut in half to expose the metal on the back side. The exposed metal had sufficient area to make good electrical contact and electropolishing was carried out easily.

The conditions for electropolishing were 0.9 amp, 35 volts, and 25 sec. in an electrolyte composed of 850 cc of ethyl alcohol, 100 cc distilled water, and 50 cc of perchloric acid.

The polished specimens were then etched in the 4 pct picral solution for a shorter time than was necessary for

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Cleveland Meeting, October 1949.

TP 2623 E. Discussion of this paper (2 copies) may be sent to *Transactions* AIME before November 15, 1949. Manuscript received January 25, 1949.

* Graduate Student Prize Paper, Seventh National Student Prize Paper Contest, AIME, 1949.

† Dept. of Physics, Purdue University.

[1] References are at the end of the paper.

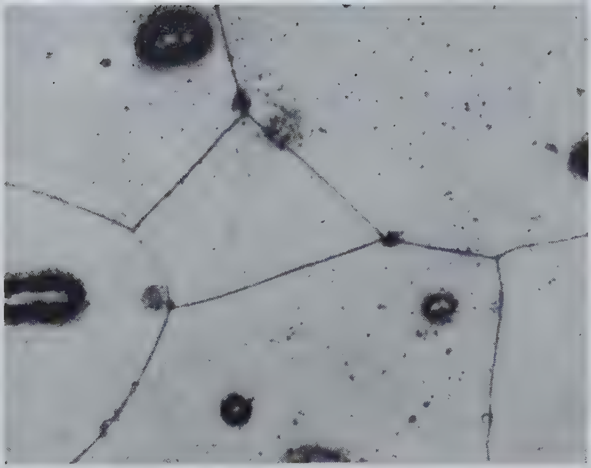

FIG 2—Ingot iron brittle. Mag. 1000 ×.
Reduced approximately one-fourth in reproduction.

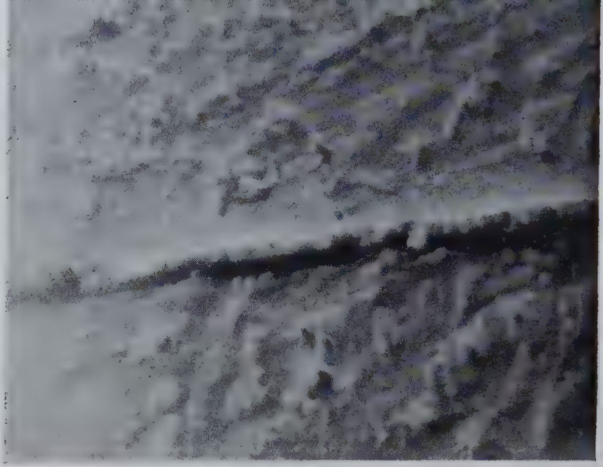

FIG 3—Ingot iron brittle. Mag. 18,500 ×.
Reduced approximately one-fourth in reproduction.

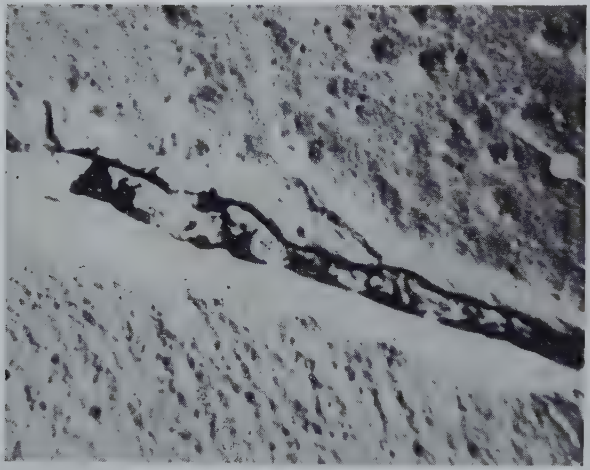

FIG 4—Ingot iron brittle. Mag. 18,500 ×.
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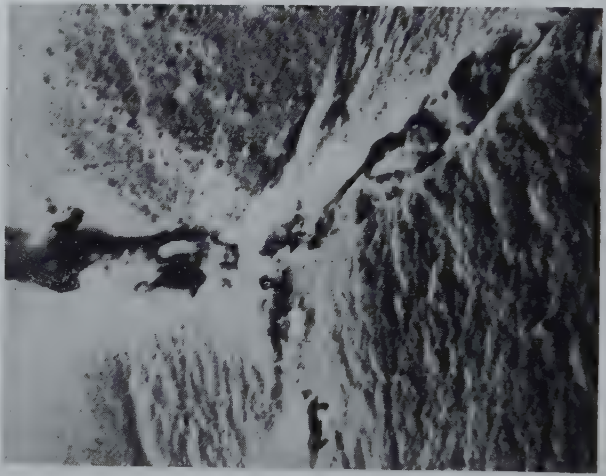

FIG 5—Ingot iron brittle. Mag. 18,500 ×.
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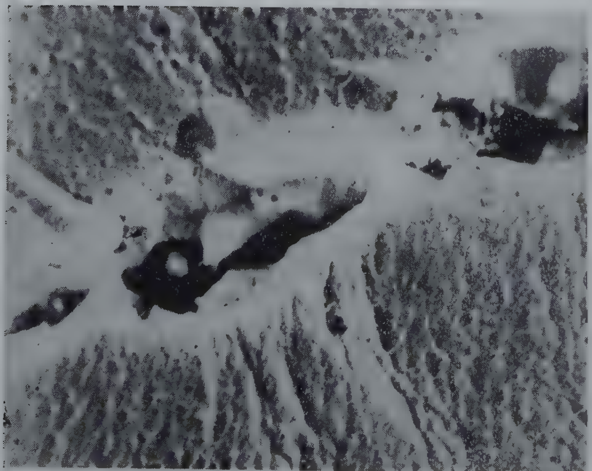

FIG 6—Ingot iron brittle. Mag. 18,500 ×.
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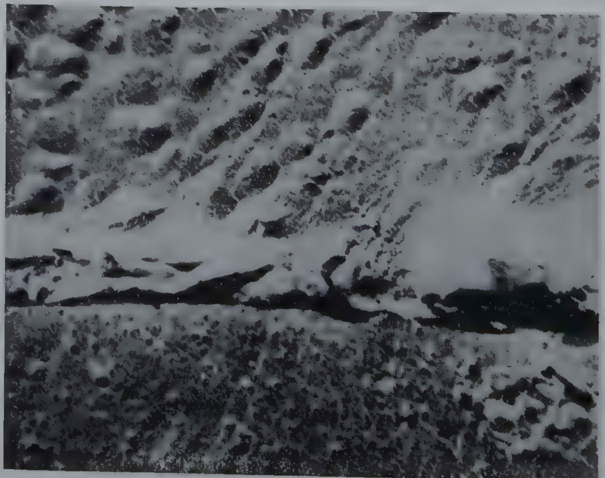

FIG 7—Ingot iron brittle. Mag. 18,500 ×.
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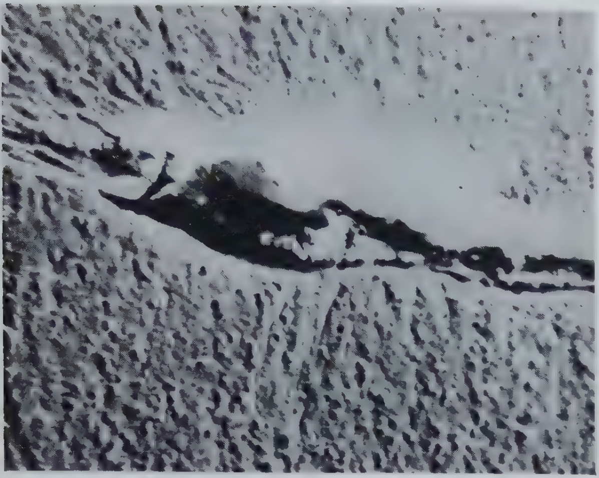

FIG 8—Ingot iron brittle. Mag. 18,500 ×.
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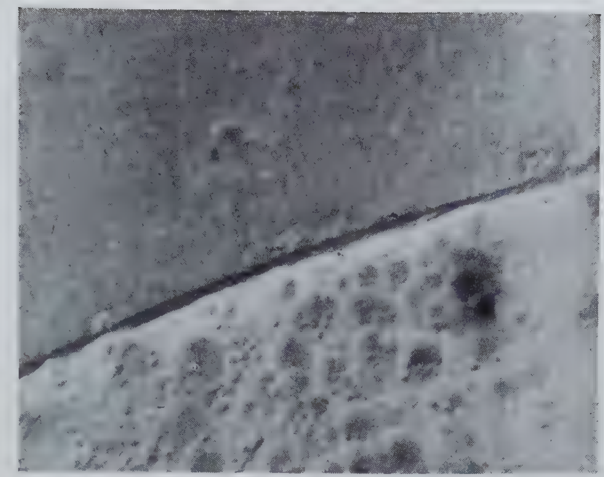

FIG 9—Ingot iron good. Mag. 18,500 ×.
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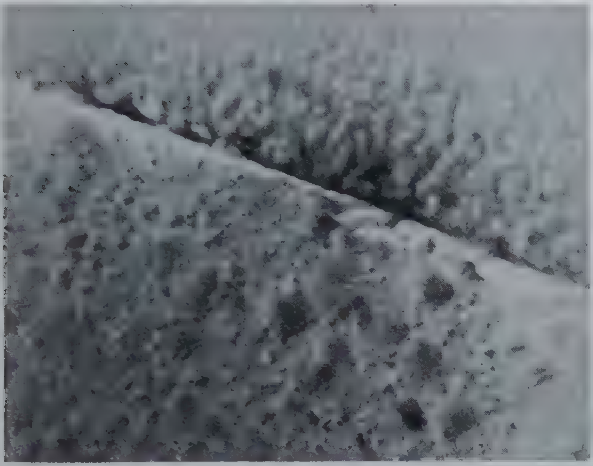

FIG 10—Ingot iron good. Mag. 18,500 ×.
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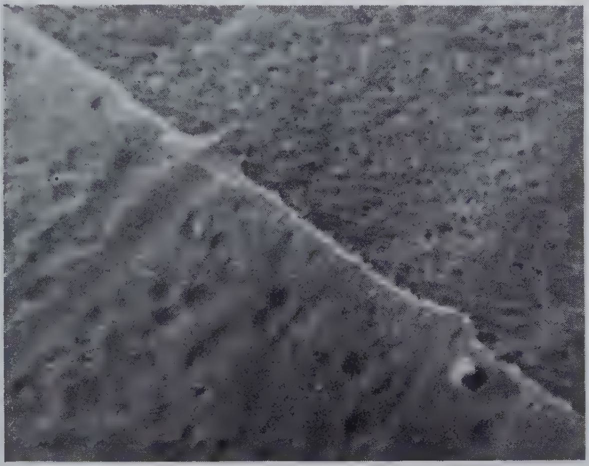

FIG 11—Ingot iron good. Mag. 18,500 ×.
Reduced approximately one-fourth in reproduction.

specimens polished mechanically. The grain boundaries were clearly defined and the number of scratches was small, Fig 2.

EXAMINATION OF THE GRAIN BOUNDARIES

In order to gain the maximum contrast, the objective aperture was installed in the electron microscope. The background electrons were eliminated, and the electron micrographs showed good contrast. The rough surface background caused by the flowed layer as seen in Fig 3 was eliminated.

The brittle piece of ingot iron was carefully examined to locate the grain boundaries and whatever impurities the grain boundaries might contain. It was noticed that a large quantity of impurities existed at the grain boundaries. These findings were recorded on photographic plates and are represented by Fig 4–8.

The good piece of ingot iron was then similarly examined to observe the amount of impurities present. Fig 9–12 show the typical structures that were observed in the electron microscope.

The cured piece of ingot iron was then similarly examined to see the effects at the grain boundaries produced by the hydrogen anneal. Fig 13 and 14 show two representative pictures of the cured metal.

Discussion of Micrographs

The optical micrograph is included to show the grain boundaries at 1000 ×. The electron micrographs will be divided into three groups showing the grain boundary structure of each group. The electron micrographs were taken at 7500 × and then enlarged to 18,500 ×.

The electron micrographs illustrated are lacking in detail especially at the grain boundaries. This loss of detail is due to the enlargements and printing from the original plates.

BRITTLE IRON

Fig 3 is an electron micrograph showing the lack of contrast due to the rays of scattered electrons which build up a general hazy background. The impurities are shown at the grain boundaries. The white areas are shadows cast by the oblique rays of evaporated manganese. The stringers shown in the ferrite areas are probably due to the incomplete removal of the flowed layer by the etching solution.

Fig 4 was taken with the objective aperture in the electron microscope. The grain boundary is very regular and is about 0.5 microns wide.

Fig 5 shows the intersection of the three grains of ferrite. In each case the boundary is large and the impurities are clearly shown. The width of the grain boundaries is about 0.4 to 0.6 microns. The same size boundary exists in Fig 7, but the shadow cast was parallel to the grain boundary. This produced a loss of details within the shadows.

Fig 7 shows the structure of the ferrite (top) which is rough and projecting. The lower grain which is

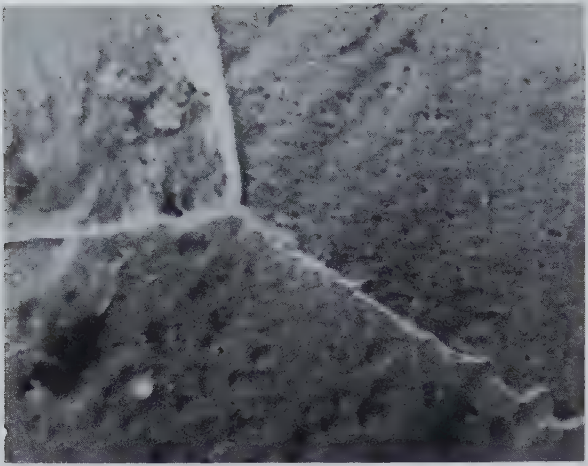

FIG 12—Ingot iron good. Mag. 18,500 ×.
Reduced approximately one-fourth in reproduction.

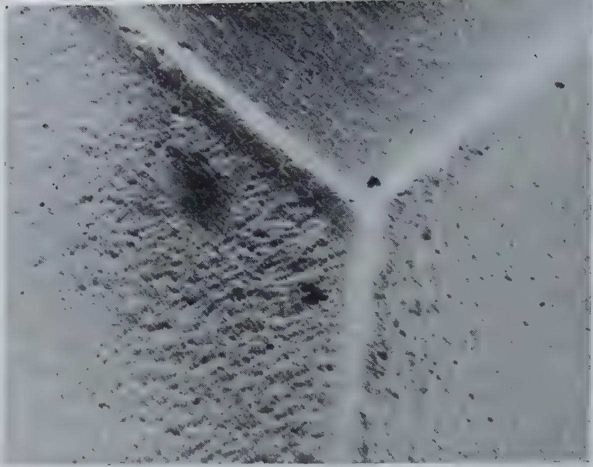

FIG 13—Ingot iron cured. Mag. 18,500 ×.
Reduced approximately one-fourth in reproduction.

oriented differently is smooth, with particles of manganese producing shadows. The grain boundaries of Fig 7 and 8 show the impurities, and the average width of the grain boundaries is about 0.5 microns.

GOOD IRON

Fig 9, 10, and 11 show the grain boundaries as narrow areas in comparison to the brittle ingot iron. The width of the grain boundaries is about 0.08 microns. However, in Fig 12 the grain boundary is wide and contains some impurities. Due to the printing process, the details within the grain boundary are missing.

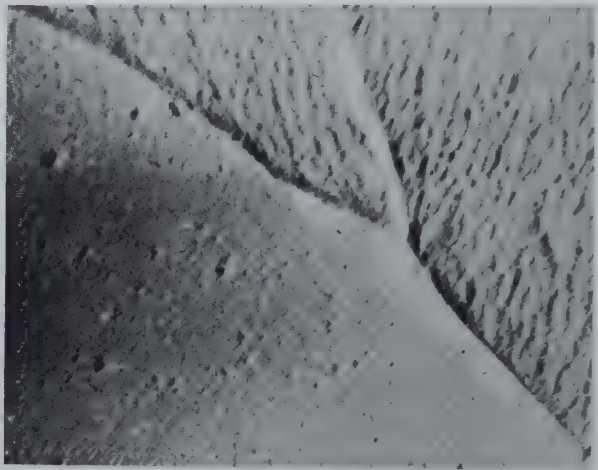

FIG 14—Ingot iron cured. Mag. 18,500 ×.
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CURED IRON

Fig 13 and 14 show the condition of the grain boundaries after the hydrogen anneal. The most noticeable fact is that the impurities have been completely removed. The grain boundaries show the grains meeting each other very tightly. The white grain boundary effect seen in Fig 13 and 14 is due also to the printing process which prohibits the fine details from being reproduced.

Fig 5 and 13 show the striking comparison of the grain boundary conditions of the brittle and the annealed ingot iron specimens.

Conclusions

From the electron microscope examination of the various pieces of ingot iron which were brittle, good, and cured, it is evident that the cause of the embrittlement may be due to the segregation of impurities at the grain boundaries. This embrittling material is probably in the form of various oxides.

In the brittle state, the grain boundaries have been broadened by the impurities which have segregated at the grain boundaries. From observation of all the plates, it has been concluded that the metal as a whole becomes brittle if the width of the grain boundaries is 0.4 microns or greater. The grain boundaries of the brittle ingot iron fall in such a range.

The good piece of ingot iron has an appreciable amount of impurities at its boundaries, but the distribution is such that the grain boundaries are not excessively large. The grain boundaries average from about 0.08 microns to 0.2 microns. It is believed that with an additional oxidizing period the good ingot iron would have gathered more impurities at the boundaries and become brittle.

The hydrogen anneal has caused the grain boundaries to give up the impurities and the bond between grains reestablished itself. There were no evidences of impurities within the grain boundaries of the cured iron. The ferrite grains were a great deal smoother and contained less inclusions than were found in the brittle and good ingot iron.

The electron microscope has proven that an actual study of the grain boundary size in various metals can shed light on many problems against which metallurgists have come. In the study of this problem, the electron microscope has shown that the grain boundaries may hold the answers to many properties of polycrystalline metal.

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Transformation of Gamma to Alpha Manganese

○ ○

E. V. POTTER,* H. C. LUKENS,* and R. W. HUBER,† Junior Member AIME

For a number of years, it has been known that manganese made by electro-deposition under certain conditions is ductile while under other conditions it is very brittle. The ductile metal is gamma manganese normally stable only between 1100 and 1138°C[1]; the brittle metal is alpha manganese, stable up to 727°C. The ductile metal is not stable, but gradually changes to the brittle form; the time required to complete the transformation is about 20 days at room temperature. Other observations have indicated that the transformation is completed in 10 to 15 min. at about 125°C, while at −10°C, no appreciable change occurs in 9 months.

The properties of gamma and alpha manganese in the pure state are ordinarily difficult to determine because the gamma structure cannot be retained by normal quenching procedures and alpha manganese is so brittle, it is difficult to obtain specimens free from flaws. In a recent investigation[2] some properties of gamma and alpha manganese were determined by studying the ductile electrolytic metal and determining the changes in its properties as it transformed to the brittle alpha form. These investigations provided an excellent opportunity for following the progress of the transition and studying its mechanism. The results of a series of such investigations are reported in this paper.

Procedure

Various properties of manganese were determined starting with the metal in the original ductile gamma form and following the subsequent changes in its properties as the metal transformed to the brittle alpha form. These observations were made at various temperatures, the data providing information regarding the mechanism of the transformation as well as the effect of temperature on the transition rate. Structure and resistivity values gave the most significant results, so this paper is concerned primarily with them.

The structure was studied microscopically as well as by X ray diffraction. The resistivity was determined on strips of the metal by measuring the potential drop across a given length of the specimen. Current was passed through the specimen by wires soldered to its ends, and the potential connections were made by wires looped around the specimen near its center. The current was determined by the potential drop across a standard resistor connected in series with the specimen, the potential drop being measured on a potentiometer.

In the temperature range from room temperature to 100°C an ordinary drying oven was used to heat the specimen. This was entirely satisfactory except at 100°C, where the time required to heat the specimen was long compared to the transition time, making the initial section of the resistivity curve unsatisfactory. To overcome this limitation, at 100°C and higher a thermostatically controlled oil bath was used to heat the specimens. The block on which the specimen was mounted was plunged into the hot oil at the start of each test. The heating time was thereby reduced from 5 min. to about 6 sec, and dependable resistivity values could be obtained through 160°C. At this point the whole transition, including the warm-up time for the specimen, required only about 20 sec and it was not considered worth while trying to extend the temperature range further.

Aside from the heating problem, the problem of making a sufficient number of accurate resistivity determinations became more and more difficult as the temperature was raised. Using the manually operated potentiometer, 100°C was about as far as it was possible to go. At this temperature and above, a self-balancing photoelectric recording potentiometer was used. Its response was quite rapid, and it proved to be entirely satisfactory all the way through 160°C, where the tests were stopped because of the specimen heating problem rather than any limitation of the potentiometer recorder.

The metal used in these tests was prepared at the Salt Lake City laboratory of the Bureau of Mines. The method of preparation is discussed in a paper by Schlain and Prater.[3] The sheets were about 2⅜ by 5³⁄₁₆ in. and varied from 10 to 16 mils in thickness. They could be cut readily into pieces suitable for the various tests. X ray and microstructure determinations were made on pieces about ⅛ to ¼ in. wide and about 1 in. long, while resistivity measurements were made on strips as long as possible and about ½ in. wide. The thickness of each sheet was not uniform over all its surface. This had no bearing on the X ray and microstructure determinations, but sections as nearly uniform and free from flaws as possible were chosen for the resistivity determinations.

The gamma manganese was electrodeposited at 30°C, the time of deposition ranging from 5 to 12 hr for each sheet. Whenever possible, the tests were started directly after the metal was stripped from the cathode; otherwise the sheet was placed immediately

Cleveland Meeting, October 1949.
TP 2583 E. Discussion of this paper (2 copies) may be sent to *Transactions* AIME before December 1, 1949. Manuscript received February 17, 1949; revision received February 28, 1949.

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* Physicists, Salt Lake City Branch, Metallurgical Division, Bureau of Mines, Salt Lake City, Utah.
† Metallurgist (Physical), Salt Lake City Branch, Metallurgical Division, Bureau of Mines, Salt Lake City, Utah.

[1] References are at the end of the paper.

in an electric refrigerator and stored at about −10°C until needed. In no case was there any appreciable difference in the behavior of the fresh and stored metal.

Results

X RAY AND MICROSTRUCTURES

Microscopic examinations of the specimens were unsatisfactory. In no case could the individual grains be identified or the different phases be distinguished. The grains are apparently too small to be studied by this method. The X ray diffraction patterns gave good results, however. In each case the pattern changed from that for gamma manganese to that for alpha manganese, the relative intensities of the two patterns changing as the transition progressed. No attempt was made to determine quantitatively the relative amounts of the two phases. It is significant, however, that no beta manganese was found at any time. Since this is the intermediate phase between alpha and gamma manganese, it might be expected that the beta phase would be an intermediate step in the transformation. Either this is not the case and gamma manganese transforms directly to alpha manganese, or any beta manganese formed transforms so rapidly to alpha manganese that the amount of beta manganese present at any time is very small.

Resistivity

Resistivity measurements were made during the progress of the transformation from gamma to alpha manganese at temperatures ranging from 20 to 160°C. The results are given in Tables 1 to 11. Examination of these data shows the change in resistivity to be slow at first, increasing as the transition progresses and again slowing as the transition nears completion. This is shown clearly in Fig 1, which shows the variation of resistivity with time at 25°C. This S-shaped curve is typical of all the resistivity data as well as the hardness and density data and is apparently characteristic of this type of transition. The effect of temperature on the transition rate is shown in Table 12, where the time required for the transition to be about half completed is given for various temperatures. The time ranges from 455 hr at 20°C to about 8 sec at 160°C, decreasing by a factor of about 2 for each 10°C rise in temperature.

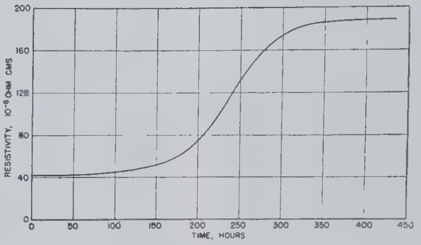

FIG 1—Variation of resistivity with time at 25°C.

Transformation Theory

Data such as those given in Tables 1 to 11 provide an excellent means for studying the transition of gamma manganese and determining the transition-rate constants, but before this can be done it is necessary to develop a theory for the mechanism of the transformation which will satisfactorily explain the observed behavior. Let us assume that the resistivity at any time is given by the expression

$$R = \alpha R_\alpha + \gamma R_\gamma \qquad [1]$$

where R is the measured resistivity, R_α and R_γ are the resistivities of alpha and gamma manganese, respectively, and α and γ are the fractions of alpha and gamma manganese present at any time. It is assumed, based on X ray observations, that no beta manganese is present. Then, since $\alpha + \gamma = 1$, the amount of alpha and gamma present at any time is given by the relations:

$$\alpha = \frac{R - R_\gamma}{R_\alpha - R_\gamma} \qquad [2]$$

and

$$\gamma = \frac{R_\alpha - R}{R_\alpha - R_\gamma} \qquad [3]$$

From Eq 2 and 3 we see that

$$\frac{d_\alpha}{dt} = \frac{1}{R_\alpha - R_\gamma}\frac{dR}{dt} \qquad [4]$$

and

$$\frac{d_\gamma}{dt} = \frac{-1}{R_\alpha - R_\gamma}\frac{dR}{dt} \qquad [5]$$

or that the rate of change of the amounts of alpha and gamma manganese with respect to time is directly proportional to the rate of change of resistivity. Thus, with only a change in vertical scale, resistivity curves such as Fig 1 also represent the variation of the amount of alpha and gamma manganese with respect to time.

From Fig 1, based on the above discussion, we see that the rate of formation of alpha manganese is slow at first, gradually increasing to a maximum near the point where half of the gamma manganese has transformed and again decreasing as the amount of untransformed gamma manganese decreases toward zero. The slow rate of formation of alpha manganese at the start, accelerating as the transition progresses, can be explained as follows: In its initial state the metal consists of atoms arranged in the form of a face-centered tetragonal structure characteristic of gamma manganese, each

Table 1 . . . Variation of Resistivity with Time at 20°C

Time, Hr	Resistivity, 10^{-6} ohm-cm	Time, Hr	Resistivity, 10^{-6} ohm-cm
0	44.9	474.3	144.3
26	45.0	505	159.7
44.5	45.4	522.8	166.6
75.8	46.0	545.8	174.1
138.5	47.0	569.8	181.9
163	48.2	597	188.0
209.5	51.5	618	192.6
239	54.8	642	198.2
260	58.3	666.8	202.1
282	63.6	690.3	204.6
306	71.8	713.8	206.2
330	79.2	739.5	207.7
355.8	87.3	767.8	209.6
379.3	93.5	786.5	210.5
403.5	103.5	815.5	211.9
427.8	114.4	858	212.5
451.3	126.09	979.5	213.8

Table 2 . . . Variation of Resistivity with Time at 25°C

Time, Hr	Resistivity. 10^{-6} ohm-cm	Time, Hr	Resistivity, 10^{-6} ohm-cm
0	42.8	240	119.3
16.8	43.1	256.5	137.4
23.8	42.4	263.3	144.1
40.5	42.5	304.5	176.5
47.5	43.0	312.3	179.4
64.3	43.1	332	183.9
71.5	42.5	336	184.2
88.3	42.8	353	186.3
95.5	44.6	357.7	186.8
136.3	49.1	376.3	187.5
144.0	50.3	400	189.0
160.5	53.9	408.3	188.6
167.3	56.2	424	187.8
184	63.7	431.8	188.8
192	68.7	472.3	189.5
208.3	83.4	480	189.2
216	92.0	496.3	189.8
232	109.3		

Table 3 . . . Variation of Resistivity with Time at 40°C

Time, Hr	Resistivity, 10^{-6} ohm-cm	Time, Hr	Resistivity, 10^{-6} ohm-cm
0	44.4	46.67	106.5
0.17	44.6	48.0	112.6
0.67	45	49.5	118.8
1.67	44.1	51	126.1
3.0	45.5	52.5	132.7
5.0	45.5	54	140.3
7.0	45.8	55	145.6
9.0	46.1	56	151.0
11.0	45.8	59.16	166.5
13.0	46.6	65	192.6
15.0	47.7	66.5	198.2
16.0	47.7	69	206.0
19.0	49.0	71	211.1
23.0	52.3	73	214.8
25.0	54.2	75	217.8
27.0	55.9	78	220.5
30.0	60.4	80	221.8
34.0	67.1	91	224.3
36.0	72.4	94.5	224.7
38.0	76.9	99.0	224.9
43.0	92.3	115	225.4
44.75	98.9		

unit cell consisting of 4 atoms. Alpha manganese, however, has a body-centered cubic structure consisting of 58 atoms to the unit cell. Before the transition can start, it is necessary that enough atoms arrange themselves in the form characteristic of alpha manganese, so that a particle of alpha manganese can form. This will occur as a result of the random motion of the atoms in the metal, the probability of enough atoms becoming arranged in the proper grouping being less for alpha manganese, however, than it would be for a more simple structure. Alpha manganese has a higher density than gamma manganese, so once a particle of alpha manganese has formed, it will try to occupy a smaller volume than the original gamma manganese, and owing to interatomic forces, the alpha manganese particle and the surrounding gamma manganese will be under stress. Alpha manganese is the lower energy form, but particles of alpha manganese below a critical size will be unstable and reconvert to gamma manganese. Particles large enough for the decrease in volume energy to exceed the increase in surface and strain energy will be stable however, and form nuclei about which more alpha manganese can form. The rate of formation of alpha manganese about any nucleus will depend on the size of the nucleus and will increase as the particle grows, new alpha manganese being formed at an accelerating rate around each nucleus. New particles of alpha manganese will also be forming throughout the body of the metal, and each of these will grow, the rate of formation of alpha manganese accelerating as the transformation progresses.

The acceleration in growth of an individual particle will continue only so long as the alpha manganese particle is completely surrounded by gamma manganese. Ultimately a point will be reached where two or more alpha manganese particles will touch each other. Their active surface is then decreased and the acceleration in growth is less. This will occur at more and more points in the specimen as the transition progresses, until a point is reached where the rate of formation of alpha manganese is a maximum and must then decrease, necessarily becoming zero when there is no more gamma manganese. Thus, curves of the general shape shown in Fig 1 can be explained on the basis of the variation with time of the interface between two phases, the interface necessarily being zero at the beginning and end of the transition and a maximum somewhere in between.

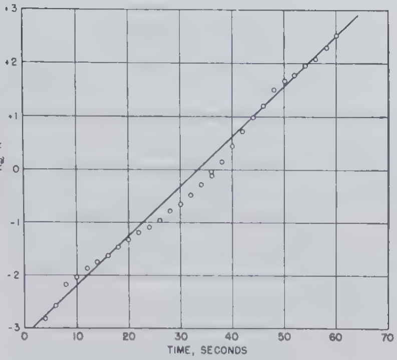

FIG 2—Log $(R - R\gamma)/(R\alpha - R)$ vs. time at 140°C.

When the particles of alpha manganese are completely surrounded by gamma manganese, the rate of formation of the alpha manganese will be proportional to the volume of the gamma manganese under stress. This volume will be the product of the interface area times an effective depth of penetration of the stress into the surrounding gamma manganese. It is reasonable to suppose that this depth of penetration will be proportional to the size of the alpha-manganese particle. Then, the effective volume of the gamma manganese under stress will be proportional to the volume of the alpha-manganese particle, or $\frac{d\alpha}{dt} = K_1\alpha$. This expression is incomplete, however, as it does not provide for a decrease in $\frac{d\alpha}{dt}$ as the alpha-manganese particles grow together and the interface area decreases. This factor cannot be accounted for in an exact manner, but the desired effect can be obtained by multiplying by gamma, as in the following equation:

$$\frac{d\alpha}{dt} = K_1\alpha\gamma \qquad [6]$$

Table 4 . . . Variation of Resistivity with Time at 58°C

Time, Min.	Resistivity, 10^{-6} ohm-cm	Time, Min.	Resistivity, 10^{-6} ohm-cm
10	44.1	502	93.9
25	45.5	517	99.1
40	46.3	532	103.0
55	46.5	562	116.2
77	46.2	592	129.2
100	47.0	624	144.9
115	47.0	657	159.5
130	46.7	725	189.5
170	48.0	779	202.7
240	50.3	804	205.8
290	53.9	826	207.7
325	57.0	854	209.8
345	59.4	889	210.6
374	62.7	914	211.1
423	71.2	950	211.6
445	76.9	1002	212.0
475	84.5	1198	212.6
487	88.1	1306	212.9

Table 5 . . . Variation of Resistivity with Time at 80°C

Time, Min.	Resistivity, 10^{-6} ohm-cm	Time, Min.	Resistivity, 10^{-6} ohm-cm
0	37.3	78	93.2
2	40.0	80	99.2
4	40.9	82	106.2
6	41.6	84	113.2
10	42.8	86	121.2
15	43.5	88	131.0
19	43.9	89	135.5
27	44.8	91	145.4
33	45.7	92	151.4
39	47.5	93	154.9
43	49.1	94	157.9
48	52.1	96	161.7
52	55.3	98	165.9
55	58.1	100	169.7
59	61.8	102	173.5
65	69.7	105	178.6
67	73.3	107	180.4
70	77.3	109	182.1
72	80.8	112	183.2
74	84.5	117	184.5
76	88.9	123	185.2

Table 6 . . . Variation of Resistivity with Time at 100°C

Time, Min.	Resistivity, 10^{-6} ohm-cm	Time, Min.	Resistivity, 10^{-6} ohm-cm
0	56.6	13	91.4
1	56.7	14	105.5
2	56.8	15	119.2
3	57.5	16	134.1
4	57.8	17	146.8
5	58.5	18	158.6
6	59.5	19	167.3
7	61.1	20	173.3
8	63.7	21	176.9
9	65.3	22	178.8
10	69.3	23	180.2
11	75.3	23.5	180.6
12	82.9		

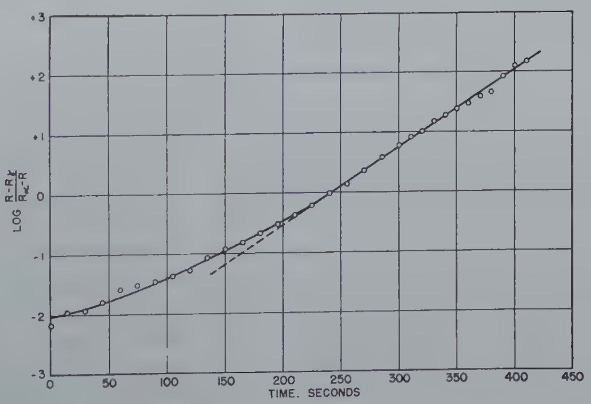

FIG 3—Log $(R - R\gamma)/(R\alpha - R)$ vs. time at 115°C.

Table 7 . . . Variation of Resistivity with Time at 115°C

Time, Sec.	Resistivity, 10^{-6} ohm-cm	Time, Sec.	Resistivity, 10^{-6} ohm-cm
0	64.3	240	125.2
15	64.9	255	135.9
30	65.0	270	152.0
45	65.5	285	162.8
60	66.6	300	171.2
75	67.1	310	175.3
90	67.7	320	178.9
105	68.8	330	181.4
120	69.9	340	183.5
135	73.6	350	184.9
150	76.3	360	185.9
165	80.1	370	186.7
180	85.5	380	187.3
195	92.0	390	188.5
210	100.5	400	189.0
225	110.7	410	189.2

Table 8 . . . Variation of Resistivity with Time at 130°C

Time, Sec.	Resistivity, 10^{-6} ohm-cm	Time, Sec.	Resistivity, 10^{-6} ohm-cm
0	67.6	75	120.4
6	68.3	78	128.5
12	68.6	81	136.9
18	69.1	84	147.3
24	69.7	87	155.9
30	70.7	90	162.6
33	71.5	93	167.8
36	72.7	96	172.1
39	73.9	99	174.8
42	75.2	102	177.5
45	76.0	105	179.0
48	77.6	108	180.5
51	79.7	111	181.4
54	82.0	114	182.1
57	84.7	117	182.7
60	89.2	120	183.0
63	93.9	123	183.4
66	98.6	126	184.0
69	105.4	129	184.1
72	112.3		

Table 9 . . . Variation of Resistivity with Time at 140°C

Time, Sec.	Resistivity, 10^{-6} ohm-cm	Time, Sec.	Resistivity, 10^{-6} ohm-cm
0	74.8	32	103.7
2	75.4	34	113.9
4	75.7	36	127.3
6	75.8	38	143.2
8	76.3	40	158.7
10	76.5	42	170.4
12	77.0	44	177.7
14	77.5	46	181.4
16	78.1	48	184.7
18	79.3	50	185.8
20	80.6	52	186.2
22	82.3	54	186.8
24	83.9	56	187.1
26	86.5	58	187.5
28	91.1	60	187.7
30	96.0	62	187.9

Table 10 . . . Variation of Resistivity with Time at 150°C

Time, Sec.	Resistivity, 10^{-6} ohm-cm	Time, Sec.	Resistivity, 10^{-6} ohm-cm
0	77.7	19	127.6
4	78.3	20	141.6
7	79.0	21	153.1
9	80.4	22	161.1
10	81.2	23	167.2
11	82.5	24	172.5
12	83.8	25	176.1
13	85.8	26	178.2
14	88.5	27	179.4
15	91.9	28	180.1
16	96.0	29	180.8
17	103.4	32	181.0
18	114.1	34	181.6

Table 11 . . . Variation of Resistivity with Time at 160°C

Time, Sec.	Resistivity, 10^{-6} ohm-cm	Time, Sec.	Resistivity, 10^{-6} ohm-cm
0	83.2	7	111.9
1	84.5	8	144.3
2	85.1	9	176.8
3	86.9	10	187.4
4	88.2	11	188.3
5	91.9	12	188.9
6	97.0	13	189.6

Thus, in the early stages of the transition, when gamma is nearly 1, Eq 6 says that $\frac{d\alpha}{dt} = K_1\alpha$ as was reasoned above. However, as alpha increases, gamma decreases, and $\frac{d\alpha}{dt}$ increases until alpha equals gamma or the transition is half complete. After this the decrease in gamma causes $\frac{d\alpha}{dt}$ to decrease, approaching zero as the transition nears completion. Introducing gamma into the equation may not be strictly correct, but it does have the desired effects of reducing the rate of formation of alpha manganese as the particles grow and their surfaces meet and of reducing the rate to zero at the end of the transformation.

From Eq 6 by integration we get

$$Ln\,\frac{\alpha}{1-\alpha} = Ln\,\frac{\alpha}{\gamma} = K_1 t + C$$

and from Eq 2 and 3

$$Ln\,\frac{R - R_\gamma}{R_\alpha - R} = K_1 t + C_1 \qquad [7]$$

This equation indicates that, if the above theory is correct, values of $Ln\,\frac{R - R_\gamma}{R_\alpha - R}$ when plotted against t should follow a straight line. Using values of R from Tables 1 to 11, values of $\log \frac{R - R_\gamma}{R_\alpha - R}$ were calculated and plotted against time. Logarithms to the base 10 were used instead of natural logarithms for convenience. Typical curves are shown in Fig 2 and 3. In every case, by proper choice of R_α and R_γ, curves such as Fig 2 can be obtained and represented by a straight line if the deviations shown are not considered serious or significant. On the other hand, curves such as shown in Fig 3 can also be obtained in which the upper portion of the curve fits a straight line very well, whereas the lower portion deviates more and more toward zero time. Considering the accuracy with which the resistivities can be determined and the closeness to which the points fit a smooth curve, curves such as Fig 2 were not considered satisfactory fits and an attempt was made to modify Eq 6 so as to obtain a fit throughout the whole transition comparable in that shown in Fig 3 for the upper part of the curve.

From Fig 3, it can be seen that the slope of the actual curve, $\log \frac{R - R_\gamma}{R_\alpha - R}$ versus time, is less during the first half of the transformation than that of the straight line determined by the upper half of the curve. The deviation of the actual values of $\log \frac{R - R_\gamma}{R_\alpha - R}$ from a projected straight line fitting the upper half of the curve is very nearly proportional to $\frac{1}{\alpha}$. Therefore, by subtracting a term $\frac{K_2}{\alpha}$ from $\log \frac{R - R_\gamma}{R_\alpha - R}$, it should be possible to obtain points following a straight line throughout the whole transition. Eq 7 thus becomes

$$Ln\,\frac{\alpha}{\gamma} - \frac{K_2}{\alpha} = K_1 t + C$$

or

$$Ln\,\frac{R - R_\gamma}{R_\alpha - R} - K_2\,\frac{R_\alpha - R_\gamma}{R - R_\gamma} = K_1 t + C \qquad [8]$$

The data shown in Fig 2 were recalculated on this basis and are shown in Fig 4. It can be seen that, by proper choice of R_α, R_γ, and K_2, the data fit a straight line very well from practically zero time to completion of the transition. The deviation at zero time cannot be considered serious, as it is uncertain just when the specimen has become uniformly heated, and readings before this are unreliable.

Eq 8 seems to be a satisfactory representation of the experimental data, and from it, by differentiation, we get the relation,

$$\frac{d\alpha}{dt} = K_1\alpha\gamma\,\frac{\alpha}{\alpha + K_2\gamma} \qquad [9]$$

Comparing this equation with Eq 6, we see that a new factor, $\frac{\alpha}{\alpha + K_2\gamma}$ has been introduced. When alpha is small compared to $K_2\gamma$ this factor equals $\frac{\alpha}{K_2}$ and approaches zero as alpha ap-

Table 12 . . . Time Required for the Transition to be Half Completed

Temperature, °C	Time	Temperature, °C	Time
	Hr		Sec
20	455	115	240
25	238	130	77
40	53	140	36.5
	Min.	150	19.3
58	590	160	7.7
80	83		
100	15		

Table 13 . . . Values of Constants in Eq 8 for Various Temperatures

Temp. °C	R_α micro-ohm-cm	R_γ micro-ohm-cm	K_2	K_1 (based on time in min.)
20	241.1	43.8	0	0.0001962
25	189.8	42.6	0	0.0005475
40			0.34	0.002843
58	212.6	37.5	0.123	0.01543
80	185.8	33.3	0.220	0.1481
100	182.3	52.4	0.0501	0.5305
115	190	62	0.0279	1.832
130	184.2	66	0.0317	6.368
140	187.6	73.1	0.0582	17.77
150	181.6	76.37	0.0252	28.55
160	190	83	0.0767	101.8

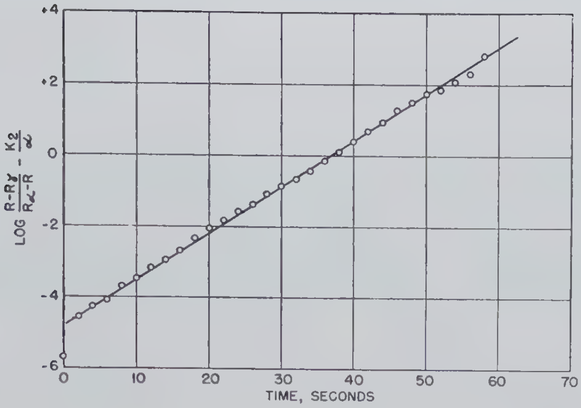

FIG 4—Log $(R - R\gamma)/(R_\alpha - R) - K_2 \frac{R\alpha - R_\gamma}{R - R_\gamma}$ vs. time at 140°C.

proaches zero, making $\frac{d\alpha}{dt}$ smaller during this part of the transition. The factor $\frac{\alpha}{\alpha + K_2\gamma}$ increases as alpha increases, becoming ½ when alpha equals $K_2\gamma$, and is equal to 1 when alpha is large compared to $K_2\gamma$. In the latter case Eq 9 is equivalent to Eq 6, and represents the upper part of the curve in Fig 3. This equation was obtained empirically and has no obvious physical interpretation. Its exact form cannot be considered too significant. A number of equations could be obtained which might represent the experimental data as well. One of these is

$$\frac{d\alpha}{dt} = K_1\alpha\gamma(1 - e^{-\frac{\alpha}{K_2\gamma}}) \qquad [10]$$

In this case, the factor $(1 - e^{-\frac{\alpha}{K_2\gamma}})$ is equal to $\frac{\alpha}{K_2}$ as alpha approaches zero, increases to 0.632 when $\alpha = K_2\gamma$, and becomes 1 when α is large compared to $K_2\gamma$. This is almost exactly the way the factor $\frac{\alpha}{\alpha + K_2\gamma}$ in Eq 9 varies with alpha. Again, however, this factor has no obvious physical significance, and in addition Eq 10 cannot readily be integrated and the constants cannot readily be evaluated from the experimental data. The important thing to be noted is not the exact form of the modifying factor but rather the manner in which it affects the value of $\frac{d\alpha}{dt}$ as alpha varies.

The physical nature of the factors retarding the transformation in the initial stages is not known. It is probably due to the greater tendency for small alpha-manganese particles to revert to gamma manganese than for the larger ones. This would slow the net formation rate of alpha manganese at first. Formation of beta manganese might also be a factor, but it is hard to see how this could have a great effect, since more than a few percent of beta manganese could hardly be present without being detected and such small amounts could hardly affect the transition appreciably.

The transformation theory presented above is necessarily greatly simplified in order to facilitate mathematical treatment, and a number of factors influencing transformations in metals have not been considered. It is to be noted, however, that this simple theory fits the observed experimental data very well for the latter half of the transition and with a relatively simple modification can be made to fit throughout the whole transition. The data in Tables 1 through 11 are included for the benefit of those who might wish to reinterpret the data.

Transition Rates

Based on Eq 8, values of log

$$\frac{R - R_\gamma}{R_\alpha - R} - \frac{K_2(R_\alpha - R_\gamma)}{(R - R_\gamma)}$$

were plotted against t as in Fig 4 for all the data in Tables 1 to 11, inclusive, values of R_α, R_γ, and K_2 being chosen to give the best straight line. This is not difficult, since the best values of R_α and R_γ can be obtained very closely from the original data and R_γ is not influenced at all by K_2. R_α and K_2 are somewhat interdependent but not seriously so, since R_α can be approximated very closely from the original resistivity data. The values R_α, R_γ, K_1, and K_2 thus obtained are given in Table 13.

The values of R_α and R_γ are quite interesting. With only two exceptions, the values of R_α fall between 180 and 190 micro-ohm-cm. R_γ varies considerably but the values up to 58°C indicate an average value of 44 micro-ohm-cm. The variation of R_α with temperature is small, indicating a low temperature coefficient, while the variation of R_γ with temperature indicates a high temperature coefficient in the order of 60 to 80×10^{-4}. Obviously these values are not accurate but they compare very well with observations reported in another paper.[2]

To see how the transition rate constants (K_1 in Table 13) vary with temperature, these data were treated according to the equation

$$K_1 = \frac{kT}{h} e^{-\frac{\Delta F\ddagger}{RT}} \qquad [11]$$

given by Glasstone, Laidler, and Eyring[4], where K_2 is the specific reaction-rate constant, T is the absolute temperature, $\Delta F\ddagger$ is the free energy of activation, and R, K, and h are constants. From this relation,

$$\text{Log}\frac{K_1}{T} = \text{Log}\frac{k}{h} - \frac{1}{2.3026}\frac{\Delta F\ddagger}{R}\frac{1}{T}$$

Values of Log $\left(\frac{K_1}{T}\right)$ were calculated and plotted against $\frac{1}{T}$. These values are shown in Fig 5. The points follow rather closely to a straight line, the greatest deviation being shown by the 160°C point. Considering the rapidity of the transition at this temperature

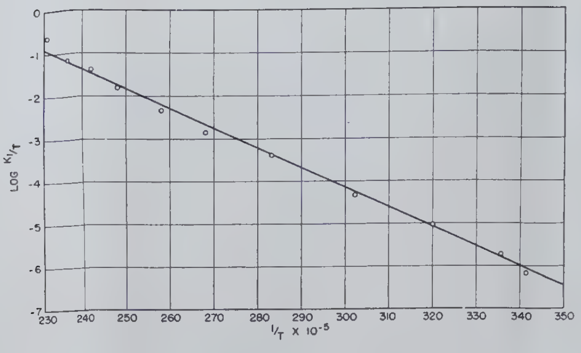

FIG 5—Log (R_1/T) vs. $1/T$.

and the time required to heat the specimen, this value may be considerably in error and its deviation is not necessarily significant. As a whole, the points fit a straight line satisfactorily, and from the slope of this line the free energy of activation was calculated to be 21,200 cal per mol.

Summary and Conclusions

The change in structure and resistivity of electrolytic manganese as it transformed from ductile gamma to brittle alpha manganese was determined. The values were found to follow a typical S curve when plotted against time. The rate of change was small at first, increased to a maximum as the transition was about one-half complete, and then decreased as the transition neared completion.

Resistivity determinations proved to be the most satisfactory way to follow the transition, and studies were made at temperatures from 20 to 160°C. It was found that the transformation curve during the latter half of the transformation could be represented by an equation based on the theory that the rate of formation of alpha manganese was determined by the amount of interface between the alpha and gamma manganese or

$$\frac{d\alpha}{dt} = K_1\alpha\gamma$$

The observed rate of formation of alpha manganese at the beginning of the transition was smaller than this theory predicted, however, and a modification of this equation

$$\frac{d\alpha}{dt} = K_1\alpha\gamma\,\frac{\alpha}{\alpha + K_2\gamma}$$

was found to represent the data quite well throughout the whole transition. The physical significance of this additional factor is not obvious. Apparently there are some factors operating in the initial stages of the transformation which retard the formation of alpha manganese but become less active as the amount of alpha manganese increases and are negligible during the latter half of the transition. This effect probably arises from a tendency on the part of the alpha manganese to revert to gamma manganese when the alpha-manganese particles are small, this tendency becoming less and less as the alpha-manganese particles grow in size.

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CORRECTION MAY ISSUE

Discussion of "The Room and Elevated Temperature Properties of Some Sand Cast Magnesium-base Alloys Containing Zinc." On page 329, column 1, line 8, of May 1949 *Journal of Metals Transactions*, discusser should be N. S. SPENCE.

Liquid Solubility of Manganese in a Magnesium-aluminum-tin Alloy[†]

B. J NELSON,* Junior Member, and G. F. SAGER,* Associate Member AIME

Magnesium alloy forgings offer higher and more uniform mechanical properties than heat treated magnesium alloy castings and are used principally for light weight parts that may be stressed in fatigue or subject to impact loads, or where space limitations do not permit the use of the necessarily bulkier magnesium alloy castings.

Table 1 . . . Magnesium Forging Alloys

A.M.C. Alloy	A.S.T.M. Alloy	Nominal Composition (Pct)				Typical Properties		
		Al	Zn	Sn	Mn	T.S. psi	Y.S.* psi	Elong. Pct in 2 in.
AMC57S†	AZ61X	6.5	1		0.2	42000	26000	10
AMC58S†	AZ80X	8.5	0.5		0.2	45000	30000	7
AMC58S-T5†	AZ80X	8.5	0.5		0.2	50000	34000	6
AM65S	TA54	3.5		5	0.2‡	40000	28000	12

* Yield strength is the stress at which the material exhibits a permanent set of 0.2 pct.

† Wrought alloys with the letter "C" in their designation are of controlled high purity, iron and nickel being held to less than 0.005 pct each. The same nominal compositions without guaranteed iron and nickel limits but with identical mechanical properties are available as AM57S, AM58S and AM58S-T5. The "-T5" temper is obtained by artificial aging after forging.

‡ When this investigation was started, the nominal manganese content of AM65S was 0.5 pct.

Compositions and Properties of Magnesium Forging Alloys

The nominal compositions and properties of the principal commercial magnesium forging alloys are given in Table 1.

The two that are most commonly used are AMC58S and AM65S. The former has the highest strength and is, therefore, employed for heavy duty applications. However, the forging of this alloy and of AMC57S ordinarily requires the use of slow speed hydraulic presses.

The forging alloy AM65S combines good, although somewhat lower, mechanical properties with excellent forging characteristics. This alloy can be forged on hammers, upsetters, rolls, and mechanical presses, as well as on hydraulic presses. These characteristics make it particularly useful for many applications.

Manganese Segregation in AM65S

The alloy AM65S contains 5 pct tin, 3½ pct aluminum, and a small amount of manganese, the manganese being added to insure satisfactory resistance to corrosion. Some manganese segregation was occasionally encountered in this alloy. In many applications, such segregation is of no particular consequence, but when forgings are acid dipped prior to the application of various chemical coatings, the segregated particles of manganese constituent may stand in relief. The resultant surface roughening may impair appearance and is undesirable in forgings employed in the textile industry, where very smooth surfaces are essential to avoid the catching and breaking of fine threads. An effort was therefore made to determine the cause of manganese segregation in this alloy and find a method for its elimination.

Radiographic examination of a series of cast ingot sections and extruded samples of the AM65S type having manganese contents ranging from zero to about 0.7 pct revealed no appreciable manganese segregation when the manganese was less than about 0.4 pct. A small amount was observed with a manganese content of 0.46 pct and considerable with manganese contents of 0.6 pct or higher.

Attempts to eliminate the manganese segregation by variations in the melting and casting conditions were unsuccessful and it soon became apparent that the trouble was attributable to the fact that the manganese contents that caused segregation were in excess of the amounts that would remain in solution at temperatures encountered in the melting and/or casting operations prior to the complete solidification of the ingots.

A knowledge of the amounts of manganese that would be soluble in molten alloys of the AM65S type at various temperatures was obviously desirable. The liquid solubilities of manganese in binary Mg-Mn alloys and in Mg-Al-Mn and Mg-Zn-Mn alloys have been determined by Tiner and others,* but no data on the solubility of manganese in molten magnesium alloys containing tin and aluminum are given in the literature. For this reason, an investigation to determine the solubility of manganese in a magnesium alloy containing 5 pct tin and 3.5 pct aluminum (AM65S type) was undertaken.

Cleveland Meeting, October 1949.
TP 2602 E. Discussion of this paper (2 copies) may be sent to *Transactions* AIME before December 1, 1949. Manuscript received October 29, 1948.

* Aluminum Research Laboratories, Aluminum Co. of America, New Kensington, Pa.

[†] Except for the variations in manganese content, this alloy corresponds to the American Magnesium Corporation Alloy AM65S or A.S.T.M. alloy TA54.

* Tiner: *Trans.* AIME (1945) 161, 351.

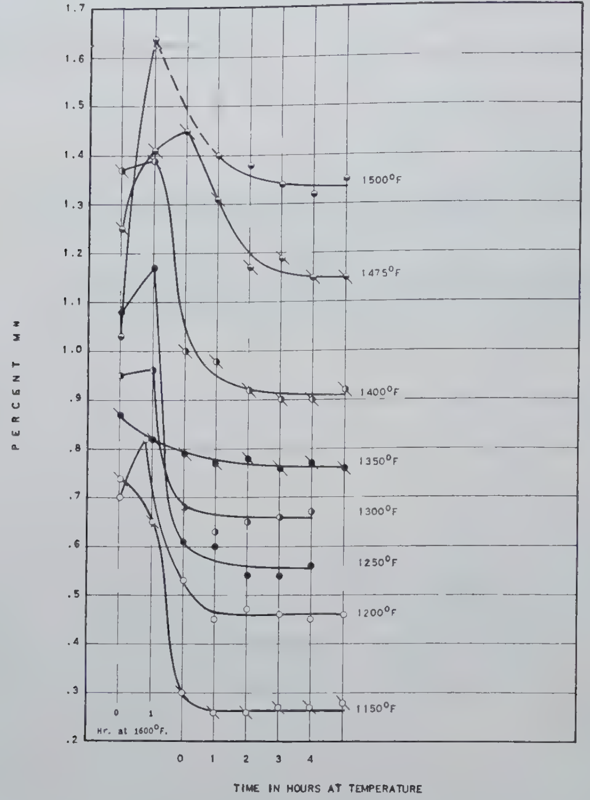

FIG 1—Manganese contents of molten AM65S as a function of holding time.

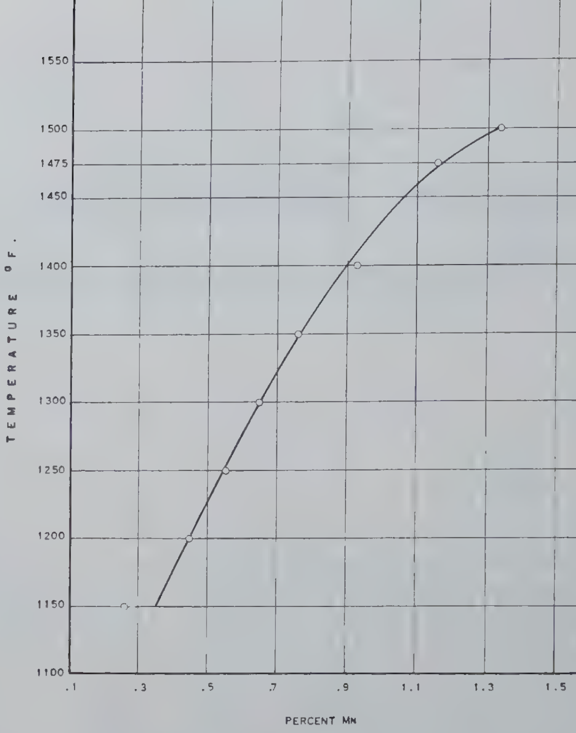

FIG 2—Solubility of manganese in molten AM65S.

Liquid Solubility Determinations

The settling procedure was selected as the most suitable method for determining the solubility of manganese in alloys of the type under consideration. Briefly, this method involved saturating the alloy with manganese at a temperature substantially above that at which the solubility was to be determined, then cooling the melt to the latter temperature and holding it at that temperature for a substantial period of time. Samples for analysis were carefully ladled from the upper portion of the melt at hourly intervals during the aforementioned holding period. After the ladling of each sample, the melt was stirred to redistribute some of the manganese that had already settled, because it appeared that when the latter particles of manganese again settled, they aided in carrying down more of the manganese and thus hastened the attainment of equilibrium.

The melts were prepared and held in a No. 8 Tercod crucible holding approximately 4 lb of metal. The manganese was added either in the form of a prealloyed ingot (Dow M) containing about 1.5 pct manganese or by the use of a flux (Dow 250) containing manganese chloride. In calculating the flux additions, it was assumed that the manganese introduced would be equal to 22 pct of the total weight of the flux. Temperatures were measured with an iron-constantan thermocouple enclosed in a seamless steel tube, the lower end of which was welded shut. This protection tube also served as a stirring rod. The samples ladled from the upper portions of the melts at the various intervals were analyzed for tin, aluminum, manganese, and iron. Table 3 shows the complete analyses for manganese, tin, aluminum and iron, together with the limiting solubilities of manganese used in drawing the solubility curve.

These manganese contents are plotted as a function of the holding time in Fig 1. It will be noted that the curves become nearly horizontal when the longer holding periods are reached, indicating that the manganese contents are approaching the equilibrium values for the various holding temperatures.

The solubility of manganese so determined at various temperatures is plotted in Fig 2. In general, the points fall on a fairly smooth curve and indicate that the solubility of manganese increases from about 0.45 pct at 1200°F to approximately 1.32 pct at 1500°F. The value for the lowest temperature (1150°F) is somewhat lower than that which would be predicted from an extrapolation of the main portion of the curve to this temperature. One possible explanation of this deviation would be the existence of a "quaternary peritectic" at a temperature between 1150 and 1200°F.

Considerable difficulty was encountered in obtaining consistent equilibrium values for the manganese solubility at temperatures near 1400°F. It was suspected that the trouble might be attributable to small variations in the aluminum contents of the alloys. In order to check this hypothesis, settling tests were conducted on alloys containing several slightly different amounts of aluminum. The results of these tests are shown in Fig 3. These results indicate that the solubility of manganese in alloys of the type under discussion decreases markedly and almost linearly with increasing aluminum contents.

While the above effect of aluminum on the solubility of manganese might be due merely to the decrease in the solubility of one element with increasing concentrations of a second solute, it was suspected that a compound containing both aluminum and manganese might be precipitating. This would, of course, result in a concentration of aluminum as well as man-

*Table 2 . . . Effect of Manganese Content on the Tensile Properties of AM65S Type Extruded and Forged Samples**

S No.	Pct Mn	Extruded†			Press Forged‡			Hammer Forged‡		
		T.S. psi	Y.S.§ psi	El. Pct In 2 in.	T.S. psi	Y.S.§ psi	El. Pct In 2 in.	T.S. psi	Y.S.§ psi	El. Pct In 2 in.
88434	0.00	36700	26700	15.0	48200	37100	7.0	34800	16900	15.7
88435	0.17	43500	33500	15.2	49400	42400	11.8	39400	26300	19.5
88436	0.26	45100	36900	14.7	49100	42800	11.8	40300	27100	18.7
88437	0.37	44900	33900	14.0	48900	43300	8.8	40900	28200	17.8
88438	0.46	45300	32800	14.0	49500	43500	11.5	39600	26600	16.8
88439	0.66	45200	31600	14.0	47800	37500	10.7	38700	25600	15.8

* All values are averages for 3 specimens.
† Tests made on standard ½ in. diam A.S.T.M. tensile specimens machined from ¾ in. diam rod.
‡ Specimens of the ¾ in. diam rod were forged down to strips $^3\!/_{16}$ in. thick. Standard A.S.T.M. sheet-type tensile specimens were machined from these strips. Both surfaces of the specimens were machined to eliminate surface imperfections.
§ 0.2 pct set.

ganese at the bottom of the melts. In order to check this point, an AM65S melt containing 1.2 pct manganese was prepared in a tapered graphite crucible. After heating to a temperature of 1550°F, the melt was cooled and held for 3 hr at a temperature of 1350°F. In order to avoid complications that might be introduced if iron dissolved from the thermocouple protection tube, temperatures were measured by a thermocouple wired to the outside of the crucible. At the end of the three-hour holding period, the crucible was removed from the furnace and cooled by a fine spray of water directed on the bottom until freezing was complete. Fig 4 shows the macrostructure of a section of the solidified melt adjoining the bottom of the crucible. The concentration of manganese constituent at the bottom is plainly evident. Analyses of the bottom zone and the main portion of the melt gave the following results:

Location	Per Cent			
	Al	Sn	Mn	Fe
Main portion....	3.17	4.82	0.50	0.001
Bottom..........	5.83	4.99	4.62	0.10

It will be noted that the amounts of aluminum, manganese, and iron in the bottom zone are substantially greater than those in the main portion of the melt.

Effect of Manganese on Tensile Properties and Resistance to Corrosion

It is obvious that manganese is likely to precipitate and segregate in AM65S melts if the manganese contents of these melts exceed the values indicated in Fig 3 for temperatures corresponding to those involved in the melting and casting of such melts. This naturally raises a question regarding the possible effect on mechanical properties and resistance to corrosion of decreasing the manganese to a value at which precipitation and segregation of that element are not encountered.

Tensile tests conducted on as-extruded, press forged and hammer forged samples of AM65S type alloys having various manganese contents ranging from zero to 0.66 pct gave the results shown in Table 2. The manganese content of the alloy can be

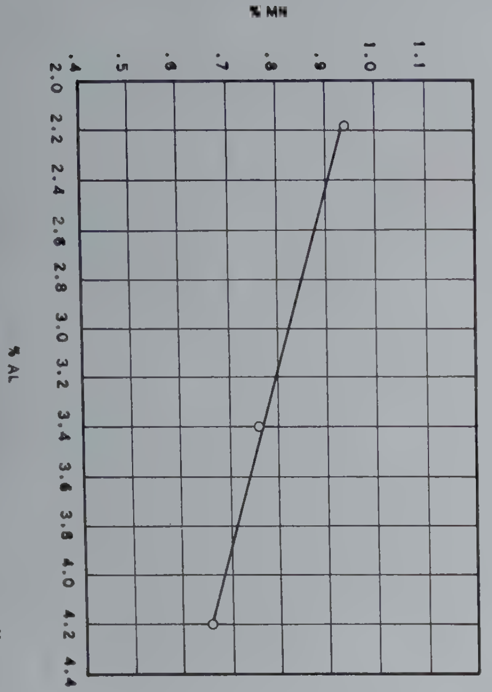

FIG 3—Effect of aluminum content on solubility of manganese in alloys of the AM65S type at 1350°C.

Table 3 . . . Summary of Analytical Data Used in Obtaining the Limiting Solubility of Manganese in Molten AM65S

Settling Temp. *T* S-No., Analytical No. Time, Etc.	1150°F 93531 1-84745				1200°F 93509 1-84745				1250°F 94065 1-85983				1300°F 94384 1-86581				1350°F 93507 1-84745				1400°F 95542 1-89009				1475°F 94841 1-87612				1500°F 94470 1-86817			
	% Mn	% Fe	% Al	% Sn	% Mn	% Fe	% Al	% Sn	% Mn	% Fe	% Al	% Sn	% Mn	% Fe	% Al	% Sn	% Mn	% Fe	% Al	% Sn	% Mn	% Fe	% Al	% Sn	% Mn	% Fe	% Al	% Sn	% Mn	% Fe	% Al	% Sn
0 hr/1600°F	0.74	0.011	3.65	5.22	0.70	0.005	2.30	3.35	1.08	0.025	3.64	5.15	0.95	0.017	3.58	4.98	0.87	0.004	1.75	2.57	1.37	0.04	3.51	5.14	1.25	0.005	3.65	5.18	1.03	0.007	3.48	5.02
1 hr/1600°F	0.65	0.018	3.57	5.27	0.82	0.015	3.57	4.96	1.17	0.006	3.56	5.25	0.76	0.020	3.54	5.04	0.82	0.019	3.68	5.12	1.39	0.016	3.44	5.14	1.41	0.016	3.72	5.25	1.64	0.018	3.50	5.13
0 hr/T	0.30	0.001	3.26	5.20	0.53	0.001	3.51	5.07	0.67	0.001	3.30	5.26	0.68	0.002	3.53	5.13	0.79	0.008	3.58	5.16	1.00	0.002	3.33	5.16	1.45	0.013	3.37	5.33	1.23	0.002	3.44	5.13
1 hr/T	0.26	0.001	3.25	5.24	0.45	0.001	3.45	5.07	0.60	0.001	3.24	5.25	0.63	0.002	2.48	5.09	0.77	0.006	3.56	5.16	0.98	0.002	3.23	5.22	1.31	0.014	3.35	5.33	1.40	0.008	3.42	5.15
2 hr/T	0.26	0.001	3.18	5.20	0.47	0.001	3.46	5.11	0.54	0.002	3.20	5.26	0.65	0.003	3.49	5.21	0.78	0.008	3.49	5.12	0.92	0.002	3.20	5.18	1.17	0.006	3.28	5.25	1.38	0.010	3.39	5.13
3 hr/T	0.27	0.001	3.12	5.24	0.46	0.001	3.42	5.11	0.54	0.001	3.18	5.26	0.66	0.005	3.52	5.28	0.76	0.007	3.46	5.22	0.90	0.002	3.20	5.24	1.19	0.011	3.32	5.33	1.34	0.005	3.35	5.19
4 hr/T	0.27	0.002	3.17	5.29	0.45	0.001	3.36	5.03	0.56	0.002	3.19	5.30	0.67	0.006	3.48	5.25	0.77	0.006	3.44	5.20	0.90	0.004	3.14	5.29	1.15	0.013	3.22	5.41	1.32	0.009	3.35	5.21
5 hr/T	0.28	0.001	3.15	5.22	0.46	0.001	3.32	5.07	0.46(?)	0.001	3.15	5.32	—	—	—	—	0.76	0.006	3.49	5.20	0.92	0.003	3.10	5.24	1.15	0.008	3.17	5.37	1.35	0.011	3.29	5.21
Limiting Solubility at *T*.	0.27				0.45				0.55				0.65				0.76				0.92				1.16				1.33			

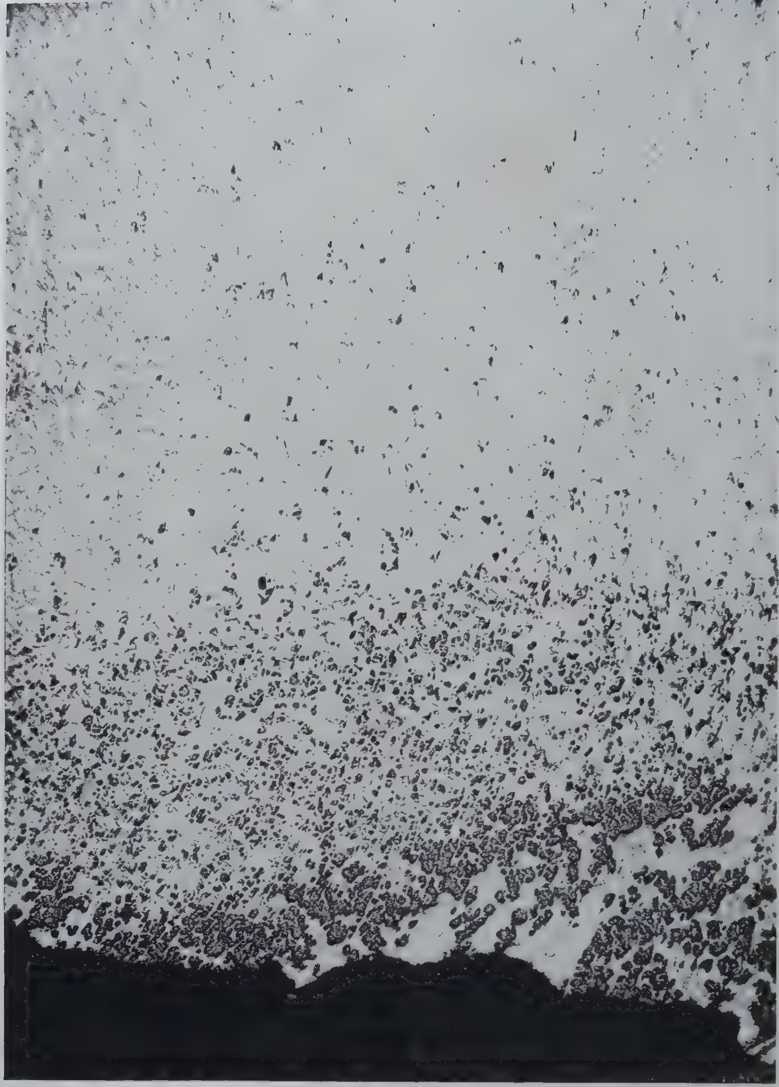

FIG 4—Macrostructure of section of solidified melt adjoining bottom of crucible.
Original mag. 10×. Etch 2 pct nital.

reduced to approximately 0.2 pct without impairing tensile properties. However, complete elimination of the manganese causes substantial reductions in the tensile and yield strengths.

Accelerated and atmospheric corrosion tests conducted on extruded samples of AM65S type alloys having various manganese contents showed that the manganese content could be lowered to about 0.2 pct without impairing resistance to corrosion. However, the complete elimination of the manganese impaired this characteristic to a significant extent.

Conclusions

1. The solubility of manganese in molten AM65S alloy increases with temperature from a value of about 0.45 pct at 1200°F to 1.32 pct at 1500°F.
2. The manganese solubility at a given temperature decreases markedly with increasing aluminum content.
3. Manganese segregation in the alloy can be controlled by maintaining the manganese content below the amount soluble at the lowest temperatures encountered in the melting and casting of the alloys.
4. The manganese content can be lowered to a sufficient extent to avoid appreciable segregation without impairing tensile properties or resistance to corrosion, but the complete elimination of the manganese would adversely affect both the strength and the resistance to corrosion.

Acknowledgment

The authors express their appreciation to the Aluminum Company of America for permission to publish this work. They also express their appreciation of the constructive guidance and advice of Dr. Wm. L. Fink, under whose supervision the investigation was carried out and to Mr. Richard Patz, who assisted in the preparation and sampling of a number of the melts.

The Comparative Creep Properties of Several Types of Commercial Coppers

A. D. SCHWOPE,* Junior Member, K. F. SMITH,* and L. R. JACKSON,† Member AIME

Table 1 . . . *Analyses of Types of Copper Used in Investigation*
(Per Cent by Weight)

Material	Tough Pitch	Tough Pitch + 15 oz Silver per Ton	Tough Pitch + 25 oz Silver per Ton	OFHC	OFHC + 15 oz Silver per Ton	OFHC + 25 oz Silver per Ton
Sb	0.0004	0.0015	0.0018	0.0010	0.0008	0.0012
As	0.0004	0.0007	0.0006	0.0004	0.0002	0.0004
Bi	None	<0.0001	None	None	0.0001	0.0001
Fe	0.0005	0.0021	0.0005	0.0007	0.0010	0.0007
Pb	0.0006	0.0005	0.0008	0.0002	0.0005	0.0003
Ni	0.0018	<0.0011		0.0011	0.0008	<0.0008
O	0.031	0.03	0.060	None	None	None
Se	0.0004	<0.0002	0.0002	0.0001	0.0003	<0.0004
Ag	0.0019	0.063	0.072	0.0011	0.0537	0.0755
S	0.0013	0.0020	0.0015	0.0011	0.0014	0.0017
Te	None	<0.0002	0.0004	None	None	<0.0006
Sn	0.0002	<0.0005		None	0.0001	<0.0001
Cu (by diff.)	99.9615	>99.8981	99.8622	99.9943	99.9411	>99.9182

Burghoff and Blank[1] have pointed out that the creep properties of hard-drawn coppers are closely associated with their individual softening characteristics and have further shown that the creep resistance of various types of copper is improved, under certain circumstances, by cold stretching.

In the design of certain types of electrical machinery, it is important to have the creep resistance as high as possible; furthermore, short-time creep strength is important since in many cases stresses are large only during warming-up and cooling-down periods.

In this paper, data are presented showing the effect of cold work on the short time creep strengths of various types of commercial copper under several conditions. It is shown that trends from short-time creep tests are evident at longer times.

Materials Used in the Investigation

The coppers used in this investigation were commercial grade and of two basic types, oxygen free high conductivity (OFHC) and tough pitch. Additions of silver in amounts of 15 and 25 oz per ton were added to each of the two coppers. The analyses of these six coppers are given in Table 1. Originally, they started as wire bars 4 × 4 × 54 in.

A square rod was made by hot rolling the wire bars to 9/16-in. rod in 8 passes. This rod was then cold drawn to 0.445-in.-diam rod and annealed for 3½ hr at 1000°F. It was then cold drawn to 0.325-in. rod and again annealed at 1000°F for 3½ hr. Hard square rods were then made by cold rolling to 0.257 × 0.257-in. rod. Soft wire was made from the hard-drawn material by annealing at 650°F for 3 hr.

The final rolling was performed using a set of 9-in. grooved rolls revolving at 30 rpm, which produced square wires. Reductions of approximately 5, 20, 30, and 40 pct were obtained using several passes in the same direction for the large reductions.

The 5 pct reduction by drawing was accomplished using a steel die having 2 parallel bearing surfaces. The die angle was 7½ degrees and the wires were lubricated with a paste of ceresin wax dissolved in carbon tetrachloride. The speed of drawing was 13 fpm. Since work was done on only two sides of the wire simultaneously, it was necessary to rotate it 90 degrees each pass to insure uniform deformation.

The room-temperature tensile properties of the various tempers are given in Table 2. The grain size of the annealed 0.257-in. wires was uniform and averaged about 0.035–0.045 mm.

Experimental Procedures for Short-time Creep Tests

Standard creep-test frames (Fig 1) were used for the testing. Special fixtures were designed to apply axial compressive and tensile loads to the copper wires. The compression specimens were 1 in. long and the ends were ground flat and parallel. The creep in compression was indicated by the movement of reference marks scratched on intersliding platinum strips attached to the moving heads of the fixture. The movement was measured with an optical micrometer fitted with a filar eyepiece on which the smallest division was 0.00005 in.

The tensile creep specimens were 4 in. long which provided ample gripping surface for the jaw-type grips. The platinum strips were attached to a one-inch gauge length of the wire and the creep measured as previously described.

The single-specimen furnaces were connected to individual power and control sources. The controlling equipment consisted of Foxboro controllers

Cleveland Meeting, October 1949
TP 2605 E Discussion of this paper (2 copies) may be sent to *Transactions* AIME before December 1, 1949. Manuscript received January 3, 1949.

* Research Engineers, Battelle Memorial Institute.
† Supervisor, Battelle Memorial Institute.

[1] References are at the end of the paper.

specially fitted with anticipating thermocouples which controlled the temperature to less than 3°F of the control setting which was 572°F (300°C). Two thermocouples were mounted on each specimen, one to the controller and the other to the recorder and potentiometer for measuring the temperature.

The specimen was mounted in its fixture ~~and~~ placed inside the furnace and assembled in the test frame. The furnace was brought up to temperature, with a zero load on the specimens, in approximately 30 min. A no-load reading was taken and the load applied by means of dead weights on the lever arm. Readings were taken thereafter at suitable intervals and the test continued until a secondary or minimum creep rate was well established. The tests averaged twenty-four hours in duration.

Table 2 . . . Room-temperature Tensile Properties of Wires

Material	Temper, Pct Cold Reduction	Yield Strength, psi*	Tensile Strength, psi	Elongation, Pct in 10 in.
Tough pitch	Drawn 5	17,500	33,400	34
Tough pitch	Rolled 5	17,500	33,500	33
Tough pitch	Rolled 20	43,400	45,700	3.5
Tough pitch	Rolled 30	46,500	47,200	3.0
Tough pitch	Rolled 40	49,000	50,200	2.5
Tough pitch + 15 oz Ag per ton	Rolled 5	24,000	36,300	33
	Rolled 20	44,000	45,300	3.5
	Rolled 30	47,500	48,800	3.0
	Rolled 40	49,500	50,700	2.5
Tough pitch + 25 oz Ag per ton	Drawn 5	18,600	35,700	34
	Rolled 5	19,300	35,900	34
	Rolled 20	43,400	45,600	3.5
	Rolled 30	47,600	48,800	3.0
	Rolled 40	51,000	52,000	2.5
OFHC	Drawn 5	14,700	32,800	36
OFHC	Rolled 5	15,000	33,000	35
OFHC	20	43,000	44,600	5.0
OFHC	30	45,800	46,900	4.0
OFHC	40	48,500	49,500	3.0
OFHC + 15 oz Ag per ton	Rolled 5	15,500	33,100	36
	Rolled 20	44,500	45,400	2.0
	Rolled 30	45,500	46,700	3.0
	Rolled 40	49,500	50,600	2.5
OFHC + 25 oz Ag per ton	Drawn 5	14,600	33,000	35
	Rolled 5	14,700	33,000	35
	Rolled 20	43,000	45,300	5.5
	Rolled 30	46,500	47,700	4.0
	Rolled 40	49,300	50,600	3.0

* Stress at 0.2 pct offset from initial slope of stress-strain curve.

Discussion of Results

The results of these tests are summarized in Table 3, which lists the creep stress, initial deformation, total creep, minimum creep rate and the total time of test. The initial deformation is seen to vary only slightly among the various coppers for similar degrees of cold work. Total creep is defined as the total strain minus the initial strain. The initiation of the third or rapid stage of creep is indicated in the table, with its approximate time.

Typical of the types of curves obtained are those shown in Fig 2 comparing various degrees of cold work. Although the hardness and the strength properties of the wires increased as the per cent reduction by cold work increased, the creep rates are not so simply affected. It is normally supposed that as the strength properties increase, the creep properties, such as rate and total deformation, are benefited. This can be considered true only as long as some competing process does not set in. As was pointed out by Burghoff and Blank, recrystallization, as manifested by softening, takes place when the strained metal is heated within a critical temperature range and the creep rate thereby increased. In a previous paper,[2] the relation between degree of cold work and the course of recrystallization for most of the coppers used in this investigation was described. It was pointed out that as the cold work increased, the temperature for softening decreased, or in isothermal annealing, the softening time decreased.

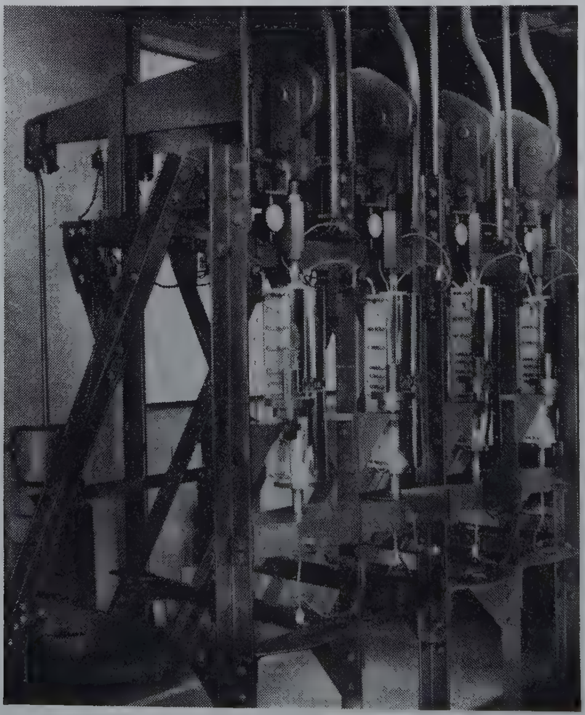

FIG 1—Creep-test Frames.

It is important, then, in analyzing creep data to keep in mind that recrystallization and creep are taking place simultaneously. Thus, in Fig 2 the curves for 5 pct cold-rolled OFHC copper appear to be normal, having progressed into the constant creep-rate stage. However, the wires rolled 20, 30, and 40 pct show decidedly different characteristics. It is evident that the beginning of the stage of rapid or acceleration creep occurs sooner as the creep stress and the degree of cold work increase.

Since softening is occurring simultaneously with creep, it is reasonable to expect the yield strength to decrease from the cold-worked value to that of soft material during testing. The majority of these creep tests were made using stresses higher than the yield strengths of the soft material so that at some time during the tests the yield stress must equal the applied creep stress. When this occurs, it is postulated that the third or final stage of creep should be initiated.

Utilizing the relations set forth in a previous paper,[2] the time for the yield strength to drop to the applied creep

Table 3 . . . Results of Short-time Creep Tests at 572°F (300°C)

Material	Temper, Pct Cold Reduction	Stress, psi	Type of Test	Initial Extension, Pct	Total Creep, Pct	Creep Rate, Pct per Hr	Total Time of Test, Min.
OFHC	Rolled 5	8,000	Comp.	0.145	0.105	0.0032	1790
OFHC	Rolled 5	10,800	Comp.	0.17	0.23	0.0074	1310
OFHC	Rolled 5	15,000	Comp.	0.24	1.12	0.025	1460
OFHC	Drawn 5	8,000	Comp.	0.145	0.120	0.0035	1580
OFHC	Drawn 5	10,700	Comp.	0.155	0.19	0.0075	1150
OFHC	Drawn 5	15,000	Comp.	0.170	1.10	0.034	1540
OFHC	Rolled 5	7,500	Tension	0.16	0.06	0.0018	1500
OFHC	Rolled 5	10,750	Tension	0.18	0.17	0.0040	1460
OFHC	Rolled 5	12,500	Tension	0.20	0.26	0.0062	1490
OFHC	Drawn 5	7,500	Tension	0.16	0.11	0.0026	1330
OFHC	Drawn 5	10,000	Tension	0.18	0.15	0.0046	1390
OFHC	Rolled 20	7,500	Tension	0.10	2.52		1445 3rd stage, 440 min.
OFHC	Rolled 40	7,500	Tension	0.15	2.59		195 3rd stage, 10 min.
OFHC	Rolled 30	12,500	Tension	0.23	8.71		725 3rd stage, 50 min.
OFHC	Rolled 20	12,500	Tension	0.12	2.61		1200 3rd stage, 390 min.
Tough pitch	Rolled 5	7,500	Comp.	0.19	0.54	0.0170	1590
Tough pitch	Rolled 5	10,000	Comp.	0.205	1.26	0.0366	1465 3rd stage, 1200 min.
OFHC + 15 oz Ag per ton	Rolled 5	7,500	Tension	0.155	0.060	0.0014	1440
	Rolled 5	12,500	Tension	0.180	0.16	0.0045	1440
	Rolled 20	7,500	Tension	0.07	0.18	0.0052	1360
	Rolled 20	12,500	Tension	0.16	0.29	0.010	1440
	Rolled 30	7,500	Tension	0.06	0.14	0.0049	1375
	Rolled 30	12,500	Tension	0.16	0.32	0.011	1355
	Rolled 40	7,500	Tension	0.04	0.16	0.0052	1440
	Rolled 40	12,500	Tension	0.17	0.31	0.0074	1440
OFHC + 15 oz Ag per ton	Rolled 20	17,500	Tension	0.265	0.680	0.023	1430
Tough pitch + 15 oz Ag per ton	Rolled 5	7,500	Tension	0.160	0.185	0.0063	1440
	Rolled 5	12,500	Tension	0.175	0.580	0.0192	1440
	Rolled 20	7,500	Tension	0.100	0.320	0.010	1395
	Rolled 20	12,500	Tension	0.170	0.700	0.0246	1415
	Rolled 20	17,500	Tension	0.295	0.765		315
	Rolled 30	7,500	Tension	0.105	0.265	0.0094	1370
	Rolled 30	12,500	Tension	0.185	0.755	0.022	1380
	Rolled 40	7,500	Tension	0.085	0.320	0.0125	1400
	Rolled 40	12,500	Tension	0.175	0.795	0.0272	1400
OFHC + 25 oz Ag per ton	Rolled 5	7,870	Comp.	0.15	0.16	0.0021	1505
	Rolled 5	10,600	Comp	0.18	0.18	0.0051	1455
	Rolled 5	12,500	Comp.	0.20	0.36	0.0089	1540
	Drawn 5	7,960	Comp.	0.16	0.18	0.0032	1430
	Drawn 5	10,700	Comp.	0.19	0.23	0.0070	1490
	Drawn 5	12,500	Comp.	0.20	0.47	0.0113	1550
	Rolled 5	7,500	Tension	0.18	0.04	0.0010	1400
	Rolled 5	10,000	Tension	0.20	0.05	0.0016	1415
	Drawn 5	7,000	Tension	0.19	0.07	0.0012	1235
	Drawn 5	10,000	Tension	0.20	0.09	0.0018	1425
	Rolled 5	12,500	Tension	0.12	0.24	0.0079	1205
Tough pitch + 25 oz Ag per ton	Rolled 5	7,500	Comp.	0.19	0.24	0.0054	1410
	Rolled 5	10,000	Comp.	0.20	0.25	0.0078	1340
	Drawn 5	7,500	Comp.	0.19	0.20	0.0060	1200
	Drawn 5	10,000	Comp.	0.205	0.42	0.0102	1340
Tough pitch + 25 oz Ag per ton	Rolled 5	7,500	Tension	0.19	0.16	0.0047	1240
	Rolled 5	10,000	Tension	0.20	0.28	0.0078	1275
	Drawn 5	7,500	Tension	0.19	0.21	0.0057	1400
	Drawn 5	10,000	Tension	0.205	0.26	0.0096	1250
	Rolled 20	12,500	Tension	0.19	0.885	0.030	1440
	Rolled 40	12,500	Tension	0.170	0.880	0.031	1340

stress can be approximated. The detailed calculations are given in Appendix A. The arrows on the creep curves indicate these computed times. Fair agreement between calculated and actual points of inflection is observed. It is to be emphasized that this relation is an empirical approximation which enables the engineer to choose a suitable degree of cold work for a particular temperature application. This relation is most effective at low amounts of cold work where the yield strength has not been appreciably changed or at temperatures where it is little affected.

Tension vs. Compression Creep

The majority of the compression creep tests covered only small ranges of strain and only in the cases where the third stage of creep set in, did local instability occur. Typical of the curves obtained are those shown in Fig 3. Tension and compression creep are compared in Fig 4 and 5.

It will be noted that the creep rate in compression is markedly higher than that for tension. In cold-drawn or cold-rolled materials, as were used here, this difference may be connected with the Bauschinger effect.

CREEP IN COLD-DRAWN VS. COLD-ROLLED MATERIALS

From Fig 4 and 5, it will be noted that in general, the cold-drawn material has a higher creep rate under comparable conditions than the cold-rolled material. This effect is probably connected with the manner in which the two methods of cold deformation distribute the cold deformation throughout the cross-section of the material. Fig 6 shows the results of a Knoop hardness transverse through drawn, rolled, and annealed tough-pitch copper. It will be noted that while the drawn material is harder at the outside, the rolled material is harder at the center. The area under the hardness curve is about 5 pct greater for the cold-rolled material than for the cold-drawn. It will also be noted from Table 2 that the yield and tensile strengths are, in general, slightly higher for the rolled than for the drawn material. These two factors probably account for the differences in creep rate observed. The nonuniformity in strain hardening evident in Fig 6 for the 5 pct cold-worked wires persists in the wires rolled 40 pct.

EFFECT OF SILVER ON CREEP PROPERTIES

It is well known that silver added to copper increases the resistance to recrystallization. Likewise, it might be expected that it should have a similar effect on the creep properties of cold-worked material. Qualitatively, this is true, but quantitatively, the relation is not simple. Fig 7 shows the recrystallization curves of the various coppers. These results are based on the fractional change in yield strength of hard drawn (84.4 pct reduction in area) 0.081-in. wire, annealed for 1 hr at various temperatures. There is little difference among the silver-bearing coppers, as far as the recrystallization curves are concerned.

The effect of silver on the secondary creep rate is illustrated in Fig 8. It is evident that silver, while effective in reducing the creep rate of both tough-pitch and OFHC coppers, does not equalize the two types as it did in the softening properties. Although the effect of silver is comparatively small at low amounts of cold working, it is very active at greater cold reductions. Its effectiveness is manifested in the delaying of the third stage of creep.

Long-time Creep Tests

During the course of this investigation, long-time tensile creep tests (>1000 hr) were performed on commercially drawn (0.081 in.) wires of tough-pitch, OFHC, and OFHC plus 15 oz of silver per ton, coppers. Three commercial tempers were included in these tests; annealed, half hard (37.1 pct red. in area), and hard drawn

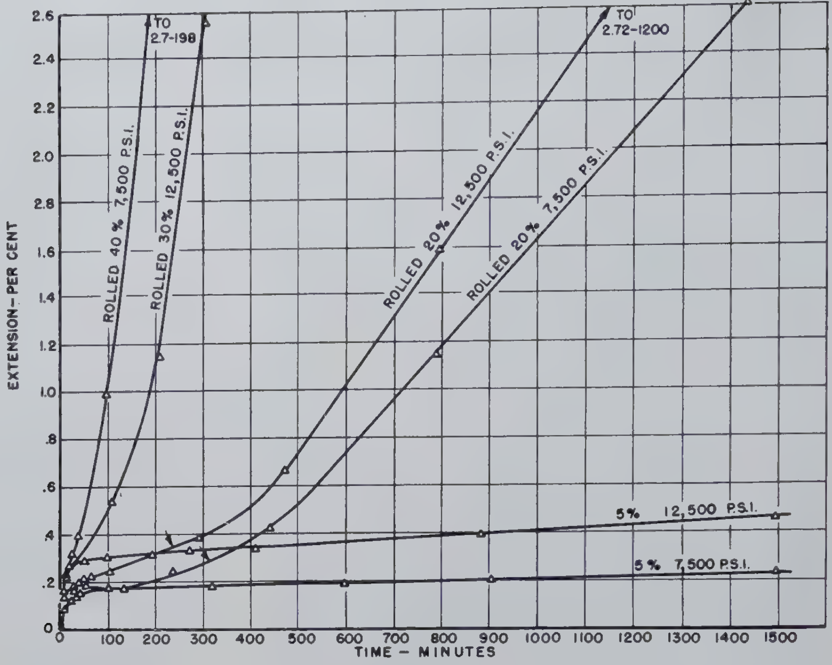

FIG 2—Time-extension curves for OFHC copper, rolled to reductions of 5, 20, 30 and 40 pct. Tested at 572°F.

Arrows indicate recrystallization has reached critical stress level.

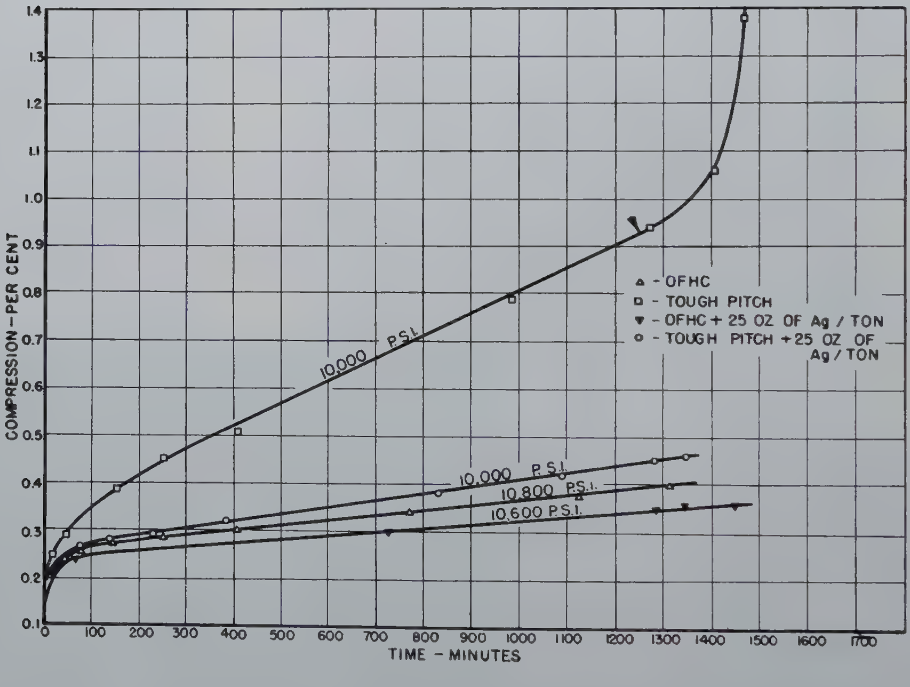

FIG 3—Time-compression curves at 572°F for various coppers cold rolled 5 pct.

(84.4 pct red. in area). An additional temper, 10 pct cold worked, was produced by pulling annealed wire in tension until the desired deformation was produced. The room-temperature tensile properties are given in Table 4.

These tests were carried out at lower temperatures and higher stresses than the short-time tests. The details of the creep tests are given in Table 5. Typical creep curves for the three coppers are shown in Fig 9, 10, and 11. The results of these tests are consistent with the short-time tests, although in some instances the effects of cold work and silver are greater than in the short-time tests.

The long-time tests serve to establish more firmly an optimum degree of cold work for each temperature and the beneficial influence of silver on the creep properties of copper. It will be observed that the long-time tests rate the various coppers in the same order as the short-time tests.

The comparative data on creep rates of OFHC and tough-pitch copper in Table 4 appear to be at variance with the results of Burghoff and Blank, who found little difference between the creep rates of some OFHC and ETP copper both in the annealed and cold-drawn condition. It should be pointed

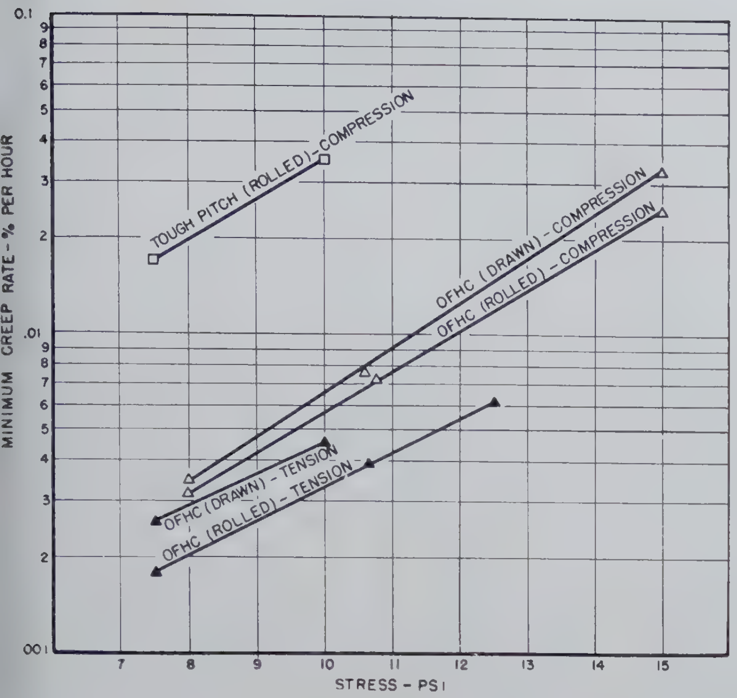

FIG 4—Stress-creep rate relationship at 572°F for coppers rolled and drawn 5 pct. Tested in tension and compression.

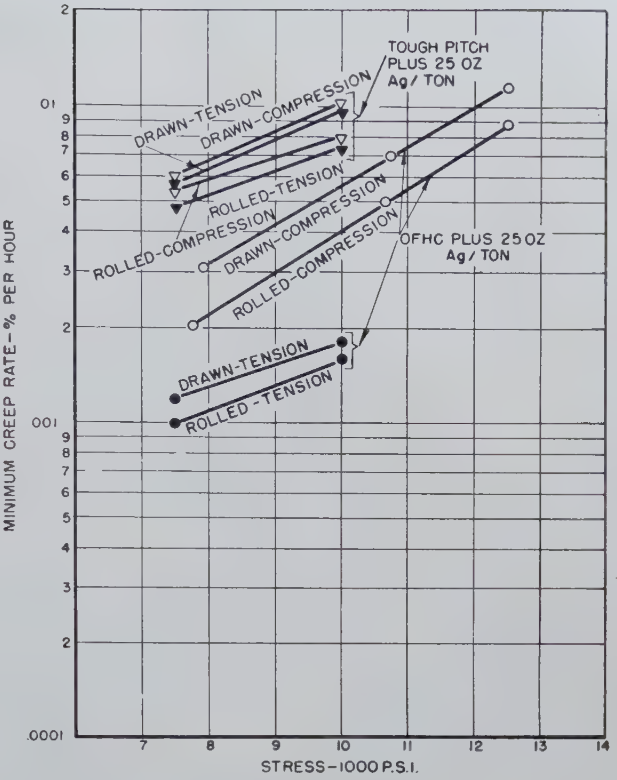

FIG 5—Stress-creep rate relationship at 572°F for coppers rolled and drawn 5 pct. Tested in tension and compression.

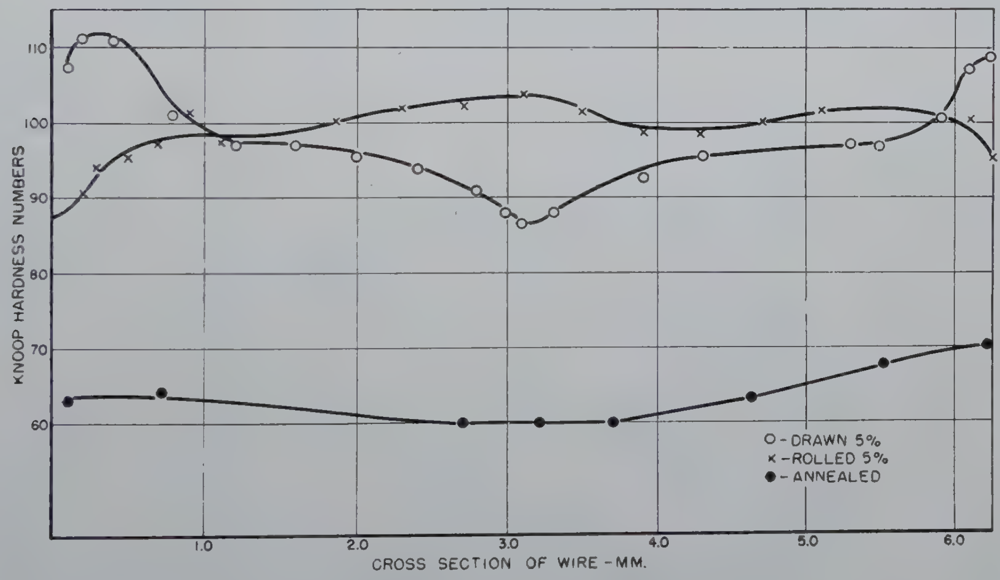

FIG 6—Knoop hardness numbers vs. location. Tough-pitch copper.

out, however, that these authors also found little difference between the softening characteristics of the two lots of copper they used in this investigation.

Summary

The effect of cold work on the creep characteristics of tough-pitch and OFHC coppers, unalloyed and silver bearing, has been determined for temperatures from 200 to 572°F. The most important results are:

1. Cold work increases the creep strength of copper; however, the benefit from cold work is lost at temperatures where recrystallization is rapid. This temperature varies with the amount of cold work and the type of copper.

2. The addition of silver to either tough-pitch or OFHC copper raises the temperature at which rapid recrystallization occurs; the effect is approximately the same on both types of copper.

3. While additions of silver effec-

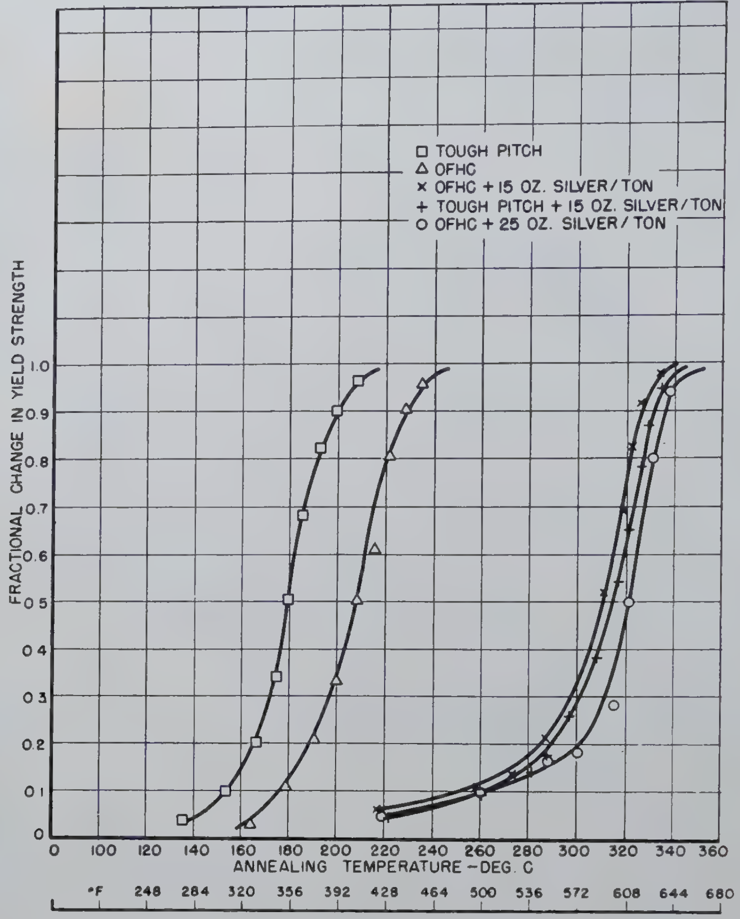

FIG 7—Softening curves for hard-drawn (84.4 pct R. A.) copper wires held one hour at temperature.

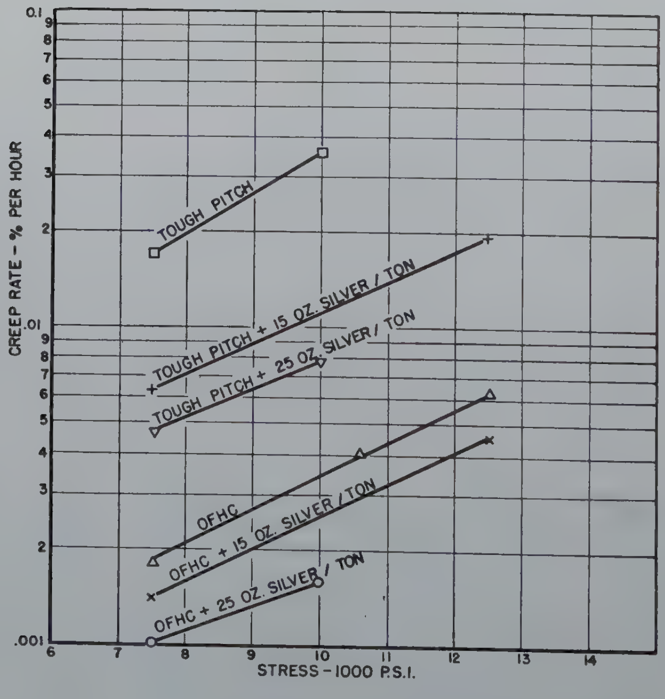

FIG 8—Effect of silver on the secondary creep rate of various coppers cold rolled 5 pct. Tested in tension. Short-time tests at 572°F.

tively lower the creep rate of both tough-pitch and OFHC copper as cold worked, the silver-bearing OFHC copper has a markedly lower creep rate than comparable tough-pitch copper.

Appendix

In a previous paper, an equation was presented by which the process of recrystallization, as evidence by softening, could be charted at low temperatures—long-time from short-time high-temperature tests. The validity of this relationship has been shown and useful curves derived by its application. The equation

$$\ln t = \ln\ln \frac{1}{1-x} - A + \frac{Q}{RT}$$

in which the terms for tough pitch copper, cold worked 5 pct, and under creep test at 300°C and 10,000 psi (Fig 3) are

t = time in hours for the yield strength to drop to 10,000 psi.

x = fraction recrystallized corresponding to a drop in yield strength to 10,000 psi.

A = a constant = 22.5 for tough pitch, taken from Fig 6 of the reference paper. (Table 6).

Q = energy of activation = 30,000 cal per mol.

R = gas constant = 1.99 cal per degree per mol.

T = temperature, degrees Kelvin = 573°K.

This equation solved for t yields a value of 21 hr at which time the yield

Table 4 . . . Tensile Tests on Copper Wires

Material	Cold Reduction After Last Anneal, Pct	Yield* Strength, psi	Tensile Strength, psi	Elongation, Pct in 10 In.
Tough pitch	84.4 (hard)	65,000	65,200	1.5
Tough pitch	37.1 (half hard)	55,000	57,000	2.0
Tough pitch	10.0	32,000	37,200	25.0
Tough pitch	Annealed	6,500	34,200	39.0
OFHC	84.4 (hard)	66,000	66,300	1.5
OFHC	37.1 (half hard)	57,000	57,500	2.0
OFHC	10.0	33,500	39,300	27.0
OFHC	Annealed	6,500	37,000	40.0
OFHC plus 15 oz per ton of silver	84.4 (hard)	65,700	65,900	1.5
	37.1 (half hard)	57,400	57,600	2.0
	10.0	33,000	38,100	27.0
	Annealed	9,000	36,100	40.0

* Stress at 0.2 pct offset from initial slope of stress-strain curve.

Table 5 . . . Results of Long-time Creep Tests

Material	Temper	Test Temperature, °F	Stress, psi	Total Creep,† Pct	Minimum Creep Rate, Pct per Hr	Duration of Secondary Stage, Hr	Total Time of Test, Hr	
Tough pitch	Hard	200	15,000	0.051	0.0000066	4500	5000	
Tough pitch	Hard	200	20,000	0.10	0.000010	3300	5000	
Tough pitch	Hard	200	25,000	0.136	0.000017	3000	5000	
Tough pitch	Hard	230	20,000	0.200			1260	Accelerating creep rate
Tough pitch	Hard	230	25,000	0.210			1000	Accelerating creep rate
Tough pitch	Soft	250	25,000	1.75			1440	Decreasing creep rate
Tough pitch	10 pct	250	25,000	1.85			2200	Decreasing creep rate
Tough pitch	Half hard	250	25,000	0.400	0.000056		4800	
Tough pitch	Hard	250	25,000	1.75			1180	Accelerating creep rate
Tough pitch	Hard	300	5,000	1.5			1100	Decreasing creep rate
Tough pitch	Hard	300	10,000	2.5			190	Accelerating creep rate
OFHC	Hard	200	25,000	0.033	0.0000049		5000	
OFHC	Soft	230	20,000	1.08	0.000042		5000	
OFHC	Soft	230	25,000	1.33	0.000067		5000	Decreasing creep rate
OFHC	Hard	230	20,000	0.046	0.0000065		5000	
OFHC	Hard	230	25,000	0.072	0.0000090		5000	
OFHC	10 pct	250	25,000	0.48	0.000060		5000	
OFHC	Half hard	250	25,000	0.240	0.000026		4800	
OFHC	Hard	250	25,000	0.167	0.000027		4800	
OFHC	Soft	300	20,000	0.800	0.00028		1000	
OFHC	Soft	300	25,000	1.4	0.00048		900	Decreasing creep rate
OFHC	Hard	300	15,000	0.4	0.00029*	260	1000	Increasing creep rate
OFHC	Hard	300	20,000	1.67	0.00058*	200	1000	Increasing creep rate
OFHC	Hard	300	25,000	1.67	0.00068*	150	680	Increasing creep rate
OFHC + 15 oz silver per ton	Soft	230	25,000	0.38	0.000025		5000	
	Soft	250	25,000	1.00	0.000090		4200	Decreasing creep rate
	10 pct	250	25,000	0.170	0.000013		5000	
	Hard	240	25,000	0.033	0.0000032		4800	
	Half hard	250	25,000	0.10	0.0000040		4800	
	Soft	300	20,000	0.750	0.00021		1100	
	Soft	300	25,000	0.880	0.00025		1100	
	Hard	300	25,000	0.066	0.000045		1000	

* Only approximate, due to extremely short second stage.

† The total creep does not include the initial elastic elongation. This quantity was not measured in these tests.

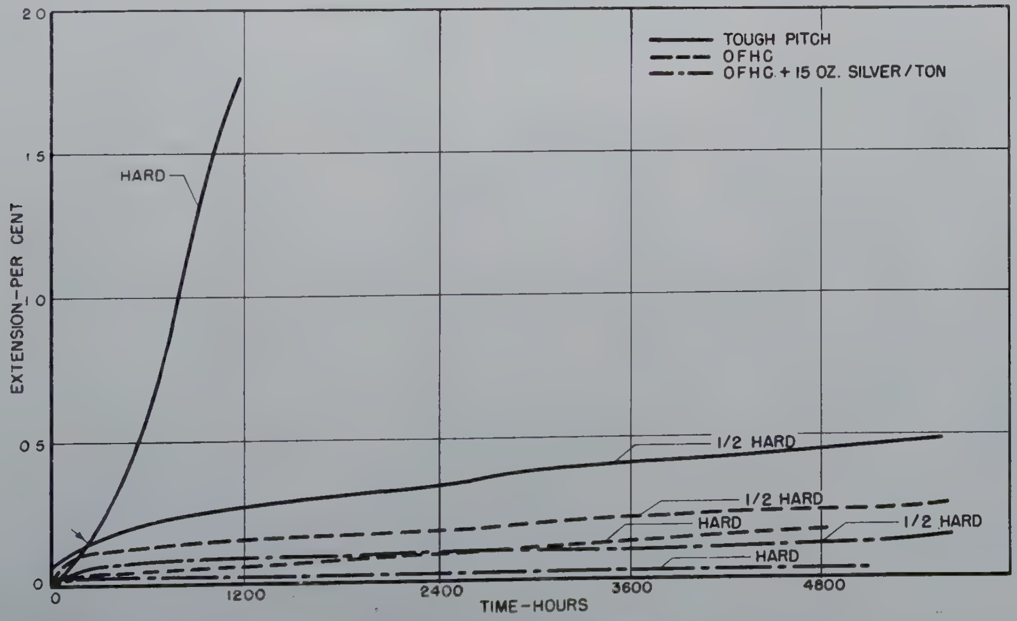

FIG 9—Time-extension curves at 250°F and 25,000 psi. Wires half hard and full hard (37.1 pct and 84.4 pct cold worked).

Arrows indicate recrystallization has reached critical stress level.

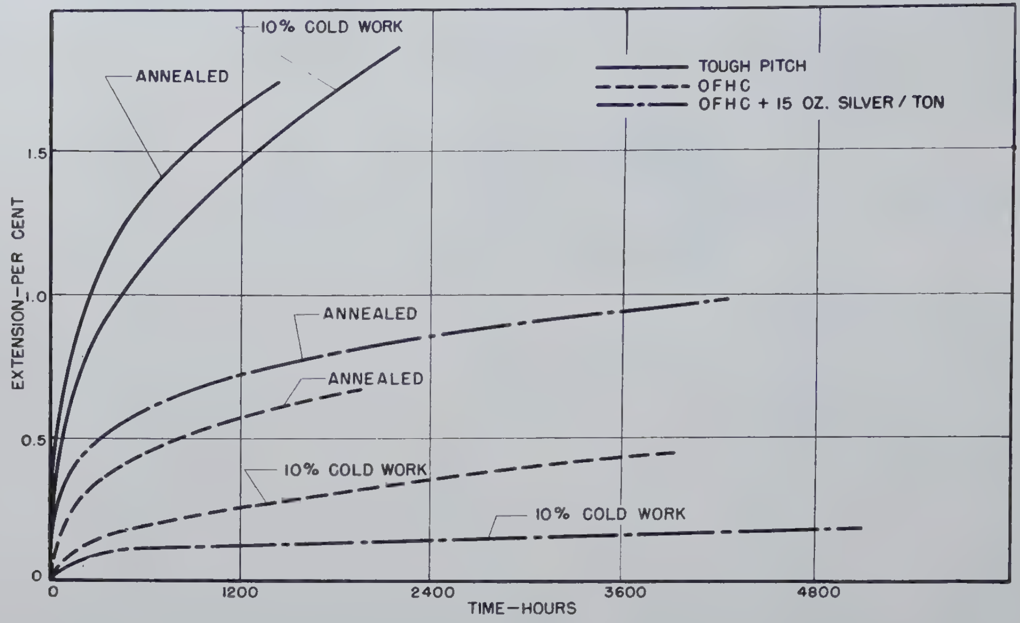

FIG 10—Time-extension curves at 250°F and 25,000 psi. Wires annealed and prestrained in tension 10 pct.

strength has dropped to 10,000 psi. At this time conditions are favorable for the initiation of the third stage of creep. When this value is placed on the curve of Fig 3 it is found to correspond closely to the inflection point. Values of *A* for the other types of copper and degrees of cold work are listed in Table 6.

Table 6 . . . Value of A for Various Types of Copper and Degrees of Cold Work

Pct Cold Reduction in Area	Tough-Pitch Copper	OFHC Copper	OFHC + 15 Oz of Silver per Ton of Copper
5.0	22.5	20.2	19.5
20.0	26.6	25.3	23.1
30.0	27.9	26.0	23.7
37.1	29.4	26.9	23.85
40.0	29.1	27.1	24.0
84.4	32.9	30.9	25.3

Considering a creep test at 25,000 psi and 300°F and neglecting the effect of strain hardening, recrystallization might be expected to reduce the yield strength to 25,000 psi for the various hard-drawn coppers in the following times: tough pitch—20 hr, OFHC—150 hr, OFHC + 15 oz silver per ton—10,000 hr. The times for the yield strengths to drop to the stress imposed during the creep tests are indicated by arrows on the time-extension curves.

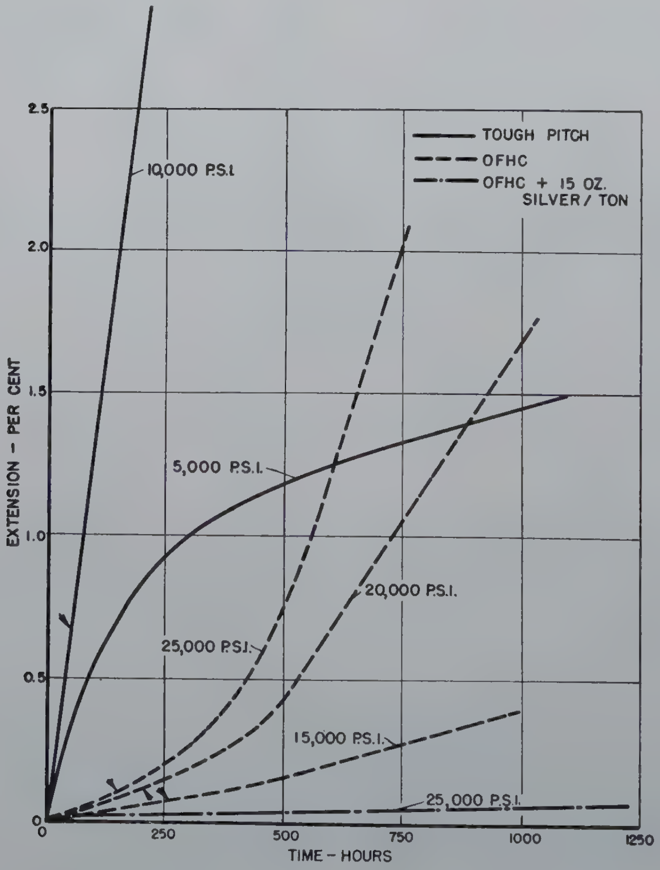

FIG 11—Time-extension curves at 300°F. Wires full hard (84.4 pct cold worked).

Arrows indicate recrystallization has reached critical stress level.

Acknowledgments

The authors are grateful to the American Metal Co. for initiating and supporting the experimental work leading to this paper. They particularly wish to thank Mr. D. L. Ogden, Mr. H. M. Burkey, and Mr. H. M. Schleicher of that company for their cooperation.

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The Rapid Determination of Orientations of Cubic Crystals

C. G. DUNN,* Member AIME and W. W. MARTIN†

Introduction

Various X ray diffraction techniques have been developed for determining orientations of crystals. Transmission and back-reflection Laue methods[1,2,3] in particular have been found to be very useful. In many applications, however, a need still exists for a simplification of the details of analysis so that orientations can be determined more rapidly, and operators with little or no training in crystallography can be easily instructed in the necessary procedures.

Filling this need in part is a system of analysis that has been in use for several years in the Metals Section of the Laboratory, General Electric Co., Pittsfield, Mass. This method is an extension of the one of Majima and Togino,[4] which uses fifty-five indexed standard transmission Lauegrams to cover the entire range of crystal orientations. Greater rapidity in determining the orientation of a crystal is achieved by using a slightly larger set of standard Lauegrams and by systematizing the steps followed. This system of analysis in its present form constitutes the subject matter of the present paper. Although the method uses transmission X ray Laue patterns, many of the operations are applicable to the analysis of back-reflection Lauegrams.

Very briefly the method is based on the fact that Bragg and azimuthal angles for two or more identified Laue diffraction spots completely determine the orientation of a crystal, and standard Laue photographs of the Majima and Togino type provide a ready means for identifying Laue spots. After a preliminary analysis, which consists of identifying the pattern in terms of one of the standard patterns, a correctly marked Lauegram is selected to carry out the final analysis. From measured Bragg and azimuthal angles, the normals (poles) of the reflecting planes for the selected Laue spots are located on a stereographic projection and the orientation of the crystal determined (in terms of the positions of cube poles for example) by a few additional operations on a Wulff net.

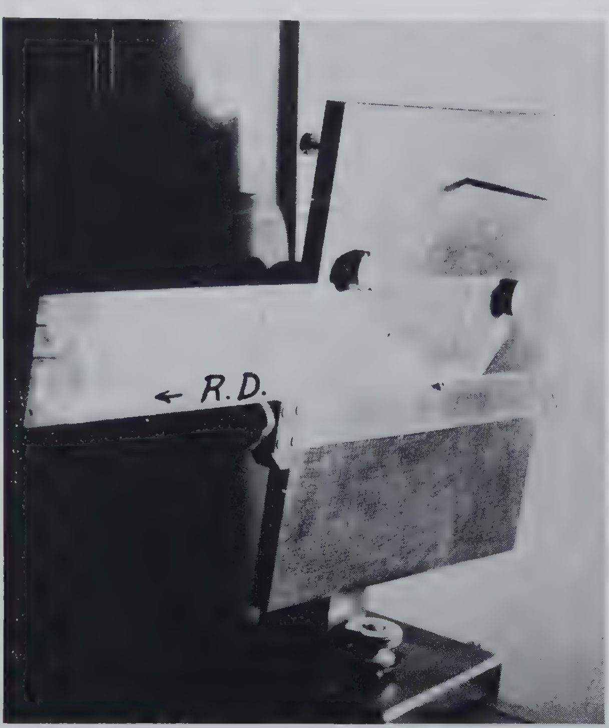

FIG 1—Photograph of a Laue transmission X ray camera with specimen in position.
Film holder not shown.

The actual procedure followed will be illustrated in detail after a brief treatment of X ray problems connected with orientation determinations and a discussion of useful principles and techniques.

X Ray and Plotting Problems

RELATIVE ADVANTAGES OF BACK-REFLECTION AND TRANSMISSION LAUE DIFFRACTION METHODS

In the back-reflection Laue method it is easy to make a direct measurement of the tilt of a zone of Laue spots. Specimen thickness also is generally no problem. In the case of thin specimens, however, the time of exposure is less for the transmission Laue method. For example, with silicon ferrite this advantage holds up to a thickness of 0.6 mm if tungsten radiation at 40 kv peak voltage is used. Furthermore, the transmission photograph represents the entire thickness of the crystal whereas the back-reflection picture represents only a surface layer (a thickness of less than 0.05 mm for silicon ferrite), the deeper layers supplying a negligible amount to the diffraction pattern. If a crystal is distorted, the transmission Laue pattern gives more information particularly with regard to the orientation of the crystal and the overall amount of spread in orientation. Finally a 3¼ × 4 in. film suffices for transmission photograms; a 4 × 5 in. or 5 × 7 in. film with a central hole is needed for back-reflection patterns Not only is the manipulation of film easier because of the size of film, but

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Cleveland Meeting, October 1949.
TP 2637-E. Discussion of this paper (2 copies) may be sent to *Transactions* AIME before December 1, 1949. Manuscript received January 21, 1949.

* Research Physicist, General Electric Co., Pittsfield, Massachusetts.

† Formerly Metallurgist, General Electric Co., Pittsfield, Massachusetts; now Metallurgist, University of California, Sandia Base, New Mexico.

[1] References are at the end of the paper.

there is also less work involved in camera loading with film requiring no central hole.

AIDS FOR THE LAUE METHOD

A convenient method for mounting flat specimens is shown in Fig 1. A collimating tube is joined centrally to a 5 in. square steel plate with the rear pinhole flush with the surface. An adjustable clamp to provide vertical and horizontal movement of the sample without rotation rests on the square plate in such a way that its upper edge is always 90° to a line passing from the rear pinhole to the long pin located near the top of the large plate (see Fig 1). The purpose of this pin is to transfer a reference direction directly to the film in the form of a pin-prick and thereby solve the alignment problem for measuring azimuthal angles. The specimen shown in Fig 1 is held in position by one clamp and two Alnico magnets. Larger specimens can be held with auxiliary clamps or with scotch tape. Further, the camera shown in Fig 1 can also be used as a specimen holder for a back-reflection camera.

RELATIONS FOR PLOTTING THE POLE OF A PLANE

The positions of poles of planes that appear in stereographic projection depend on which surface of a flat specimen is used for reference. Our procedure is to use the surface which is visible when the sample is mounted for X raying, either for transmission or back-reflection photography (as shown in Fig 1). Fig 2 diagrammatically shows the position of the normal N to a plane which diffracts X rays to a point L on the film. The normal N makes an angle δ with the surface of the sample, and the diffraction spot L makes an azimuthal angle ϕ with the reference direction, $R.D.$ The distance r from the central image O to the spot L is given by Eq 1:

$$r = D \tan 2\,\delta^{*} \qquad [1]$$

where D is the distance between specimen and film.

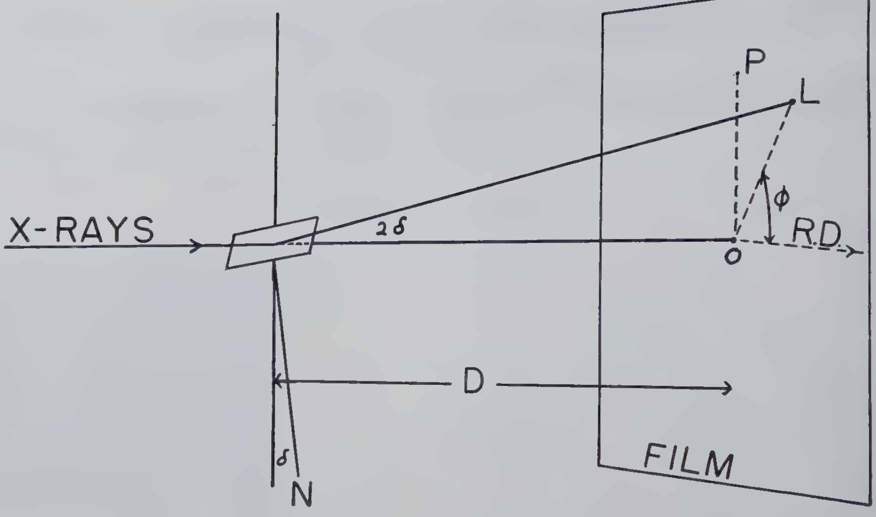

FIG 2—Schematic diagram showing angular relations for the Laue method.

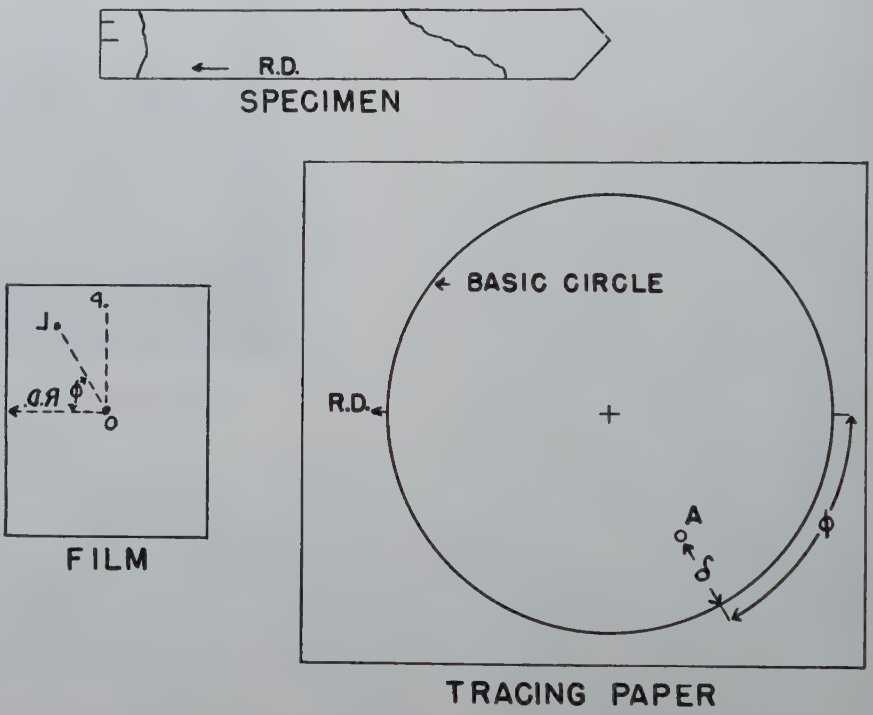

FIG 3—Schematic diagram showing correct relation for film, specimen, and stereographic projection.

The point A locates the direction of the pole of the plane which reflects X rays to the point L on the film.

The incident beam, the diffracted beam, and the normal to the reflecting plane, lie in a plane which intersects the film along the line OL at an angle ϕ to the reference direction of the sample. This relationship also determines ϕ for plotting the pole of the plane in stereographic projection. The distance r determines the value of δ according to Eq 1; therefore, a direct reading net can be easily made for measuring δ. Later a photograph will be shown of an arrangement for holding the film and measuring δ and ϕ directly (it is called a δ, ϕ analyzer).

To keep relationships correct during analysis the film is held so the observer looks through the film onto the surface of the sample. This applies to both transmission and back-reflection work. Fig 3 showing specimen, film, and the plot of one pole in stereographic projection illustrates this for transmission. As in Fig 2, the pole of the reflecting plane is diametrically opposite the Laue spot.* Its distance from the basic circle, which coincides with the plane of the sample, is δ degrees measured along a diameter. The basic circle and two diameters of a Wulff net are properly graduated for making this plot. The meridian and latitude lines of the Wulff net are also indispensable for plotting crystallographic directions from the known angular relationships between planes. Therefore, the plot is most conveniently carried out on tracing paper superimposed on a Wulff net.[5]*

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* A similar equation holds for back-reflection, the angle δ being replaced by $90° - \delta$.

* In back-reflection work the pole of the reflecting plane and the Laue spot fall on the same side of the incident beam.

In a Laue photogram certain groups of diffraction spots lie on conic sections (ellipses, parabolas, and hyperbolas). Such groups are called zones. In stereographic projection poles of planes for a zone fall on a meridian line when the plot is correctly rotated about the center of the Wulff net (in space they lie in a common plane). The direction 90° away (and this will fall on an equator when the poles of the zone are superimposed on a meridian line) is the zone axis of the zone. When a zone axis is within 45° of the center of projection, the zone of Laue spots on the film is an ellipse; when a zone axis is at 45°, the zone is a parabola; and when a zone axis is beyond 45°, the zone is a hyperbola. In back-reflection Laue photographs all zones are hyperbolas.

Crystallographic Relationships in Cubic Crystals

ANGLE BETWEEN PLANES

The angle θ between two planes $(h_1k_1l_1)$ and $(h_2k_2l_2)$ or two directions $[h_1k_1l_1]$ and $[h_2k_2l_2]$ can be calculated from Eq 2 when the Miller indices are known.

$$\cos\theta = \frac{h_1h_2 + k_1k_2 + l_1l_2}{\sqrt{(h_1^2 + k_1^2 + l_1^2)(h_2^2 + k_2^2 + l_2^2)}} \qquad [2]$$

when $h_1h_2 + k_1k_2 + l_1l_2 = 0$, $\theta = 90°$.

ZONE AXIS

Since $h_1u + k_1v + l_1w = 0$

and $h_2u + k_2v + l_2w = 0$

$$\text{when} \quad \begin{cases} u = a(k_1l_2 - k_2l_1) \\ v = a(l_1h_2 - l_2h_1) \\ w = a(h_1k_2 - h_2k_1) \end{cases} \qquad [3]$$

(where a is a suitable constant to make u, v, and w small integers) the direction $[uvw]$ is 90° from the directions $[h_1k_1l_1]$ and $[h_2k_2l_2]$, which are the poles of planes $(h_1k_1l_1)$ and $(h_2k_2l_2)$ respectively since the system is cubic. The line of intersection of the planes $(h_1k_1l_1)$ and $(h_2k_2l_2)$ is the direction $[uvw]$.

$$\text{Also} \quad h_iu + k_iv + l_iw = 0 \qquad [4]$$

when

$$\begin{cases} h_i = mh_1 + nh_2 \\ k_i = mk_1 + nk_2 \quad \text{for any integral} \\ l_i = ml_1 + nl_2 \quad \text{values of } m \text{ and } n. \end{cases}$$

Consequently, the direction $[uvw]$ makes an angle of 90° with all directions of the form $[h_ik_il_i]$ and, therefore, is the zone axis of all planes $(h_ik_il_i)$. Example: starting with Miller indices $\bar{1}$, 0, 1, 1, $\bar{1}$, and 0 for h_1, k_1, l_1, h_2, k_2, and l_2 respectively and using values of 3 and 2 for m and n respectively, we obtain [111] for the zone axis of the $(\bar{1}01)$, $(1\bar{1}0)$, and $(\bar{1}23)$ planes.*

The Relationship between Film Nets and Stereographic Projection Nets

When poles of planes for a zone are plotted in stereographic projection, points are obtained which fall on a meridian line. Also, if the direction of plotting is reversed, a meridian line of a Wulff net becomes a conic section on a film net. Let us call the conic section thus obtained, however, a zone or meridian line on a film net. Similarly a latitude line of a Wulff net can be plotted to obtain a latitude line of a film net. The reverse plotting must be carried out according to whether the net is to be used for transmission Lauegrams or back-reflection Lauegrams. Using an arrow to indicate the operation of plotting, the following relations can be indicated for well known nets.

Film Net	Projection Net	Use of Film Net	Lit. Ref.
Greninger ⇄	Wulff net	Back-reflection Laue	3b
Leonhardt zero-tilt ⇄	Wulff net	Transmission Laue	6
Leonhardt 90°-tilt ⇄	Polar net	Either B.R. or Trans. Laue	6
Leonhardt *w*-tilt ⇄	Intermediate net between Wulff and Polar net.	Transmission Laue	6

Fig 4 shows one-half of a Leonhardt zero-tilt net;[6] the dotted curves spaced at 10° intervals are meridians and the solid line curves spaced at 5° intervals are latitude lines. The distance D satisfies Eq 1, r becoming equal to D when δ equals 22½°. An illustration will be given later showing how angles between planes can be obtained with this net upon placing a zone of Laue spots on a meridian (by interpolation) and reading differences in latitude between Laue spots.

Fig 5 shows a new diagram that we have developed and which we shall call an isogonal net because each curve represents a constant angle from a point on the base line—the straight line graduated in 5° intervals. The net is a combination of tilted Leonhardt curves (w equal to 5, 15, and 25°) with no meridian lines and with two sets of latitude lines. One set gives the 45, 90, and 135° differences in latitude, the other set the 30, 60, 90, and 120° differences in latitude measured from correct points on the base line. The base line must be used for all measurements of angle between planes except those of a zone. In this particular case, as will be illustrated later, the angles between planes for a zone can be read directly when one Laue spot of a zone is placed on the base line.

The Greninger net has been adequately described elsewhere. An illustration, however, using such a net will be given later.

The film net shown in Fig 6 is very useful for plotting poles of planes and directions of zone axes in stereographic projection. When used for these purposes, however, the net must be coupled with a scale for measuring the azimuthal angle ϕ. The angle δ for any Laue spot can be read from the polar scale which is drawn in half circles. The tilt of a zone measured from the center of projection can be read directly from the net when the zone of Laue spots is correctly located by interpolation among the zones of the net. Unlike the zones of the Greninger net, however, the accuracy of measurement of tilt falls off rapidly near 90° and the position of Laue spots in the direction of the zone axis must be used for an accurate determination of the tilt of the zone.

Standard Laue Photographs

For several years Majima and Togino standards, which were reindexed for simplicity of analysis as will be described later, were used for identifying Laue spots and zones of Laue spots. Experience thus gained indicated a need for a larger set to cover all possible orientations more uniformly; so 75 patterns were made and these were prepared in two sets—one for *preliminary analysis* and the other for *final analysis*.

In all 300 patterns were made for the body-centered cubic lattice and the face-centered cubic lattice. However, it is not the purpose of this paper

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* This article, incidentally, gives an excellent treatment of the properties and uses of a Wulff net.

* Standard projections (see Ref. 1) can also be used to determine members of a zone and the zone axis.

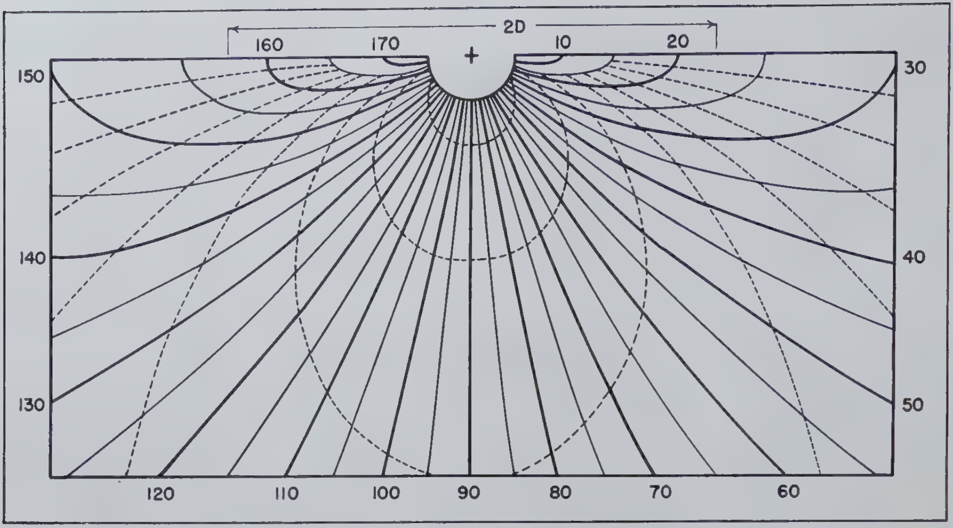


FIG 4—A Leonhardt zero-tilt net showing meridians (dotted-lines spaced at 10° intervals) and latitude lines (solid-lines spaced at 5° intervals).
Net to be used on films where film to sample distance is *D*.

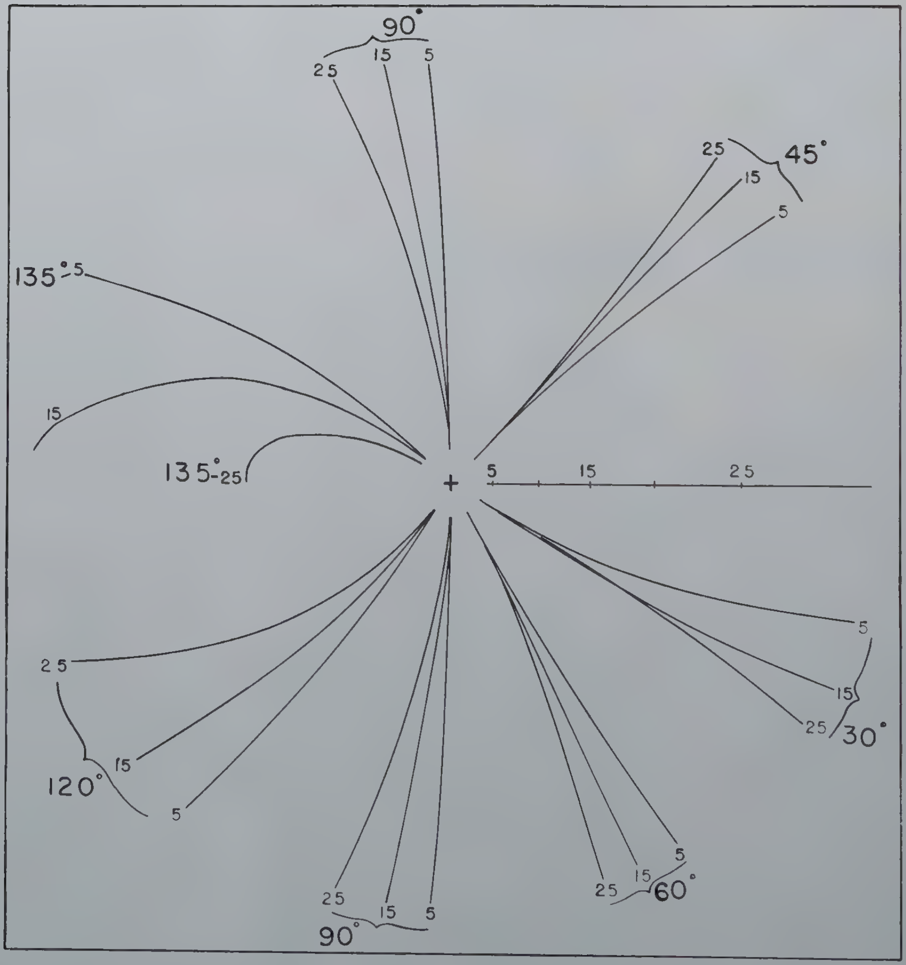


FIG 5—An isogonal film net for determining special angle relations as an aid to the identification of a Laue pattern.
To same scale as the Leonhardt net of Fig 4.

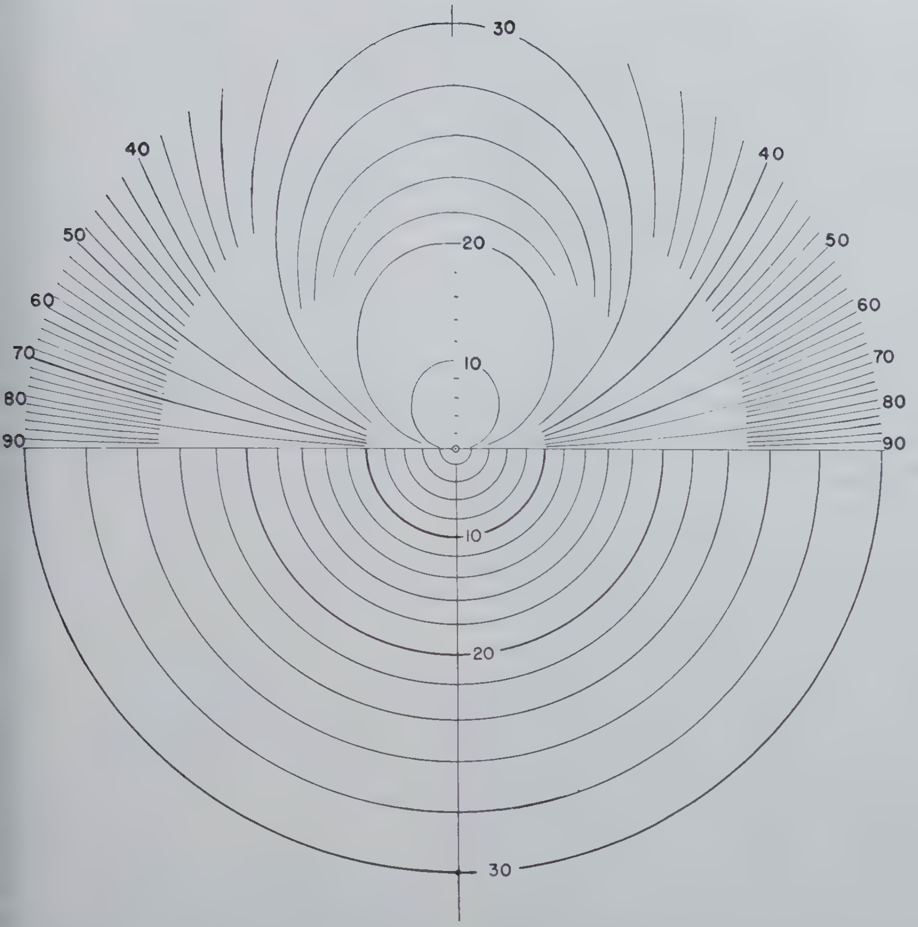


FIG 6—A film net for transmission Laue patterns when used as part of a δ, ϕ analyzer.
One half consists of latitude lines of a polar net graduated in 2° intervals; the other half consists of zones spaced at 2° intervals. To same scale as the Leonhardt net of Fig 4.

to present all 300 patterns in full size reproduction; rather the aim is to illustrate how such patterns may be used for rapid analysis.

COORDINATES OF STANDARD LAUE PATTERNS

The two angular coordinates to be considered now and discussed in relation to Fig 7a and 7b locate only the crystallographic direction that is parallel to the incident X ray beam. They are not sufficient to determine the orientation of a crystal because rotation about the X ray beam as an axis is still indeterminate.

Fig 7a shows diagrammatically the angles α and β used by Majima and Togino with their standard Laue patterns and the angles α and β' used by the present authors with a new set of standards. The relationship between β and β' is

$$\tan \beta' = \cos \alpha \tan \beta \qquad [5]$$

When α and β are zero, β' is also zero and the X ray beam is then normal to a cube plane.

The stereographic projection can be treated in two ways to show the angles α and β'. One way makes use of the unit stereographic projection triangle (see Fig 7b) formed by the [100], [110], and [111] directions of orientation A, which is plotted in terms of cube poles. The values of α and β' are zero for orientation A. Now any crystallographic direction P whatsoever may be found in the unit triangle and its position determined by two angles α and β' if we understand that the direction P is actually to be brought to the center of projection. The coordinates of P, which may be given by α and β', then specify the crystallographic direction which coincides with the incident X ray beam. Suppose P is brought to the center of projection by two angular rotations—one of α degrees about a vertical axis in the plane of the paper, the other of β' degrees about a horizontal axis in the plane of the paper. Expressed in terms of cube poles, the final orientation is C and the intermediate orientation after the α-rotation is B. On the Wulff net, α gives the change in longitude whereas β' gives the change in latitude of point P during this rotation of A to C. The unit triangle will be used again later.

The second way of determining α and β' for orientation C is given simply as follows: place the outermost cube pole on an equator of a Wulff net and read directly the angles α and β', which are determined entirely by the cube poles of orientations C and A as indicated in the figure. This relationship, incidentally, provides a quick and convenient check of the analysis made with the aid of a standard Laue pattern.

Similarly α and β may be found in two ways from the stereographic projection. First if the coordinates of P are expressed in terms of meridian intercepts on two diameters at 90° as shown, the angular distances to the center of the projection are α and β. Secondly, they can be found from the cube poles of the orientation C by constructing a diameter through the correct cube pole and locating its point of intersection in the corresponding {100} plane. Actually no diameter need be drawn when the plot is on tracing paper because the equator of a Wulff net becomes the desired diameter upon placing the correct cube poles on a meridian line.

In the unit stereographic projection triangle, a point indicates that crystallographic direction which is parallel to the incident X ray beam. Directions for the Majima and Togino set of standards, which are 0,0; 5,0; 5,5; etc. to 45,45 in α, β units, are shown in the unit triangle form in Fig 8a. Each direction falls at the center of a circle of 2° radius. These circles, forming horizontal and vertical rows, fall on meridian lines. Positions of the new standards (see Fig 8b) were chosen so that horizontal rows would fall on latitude lines and the coverage of the unit triangle would be generally both complete and uniform. Some points, which are marked with central black dots, did not fit into the otherwise regular scheme. Inaccuracies in both Fig 8a and 8b are due largely to the errors in the enlarged Wulff net, which was used to make the plots. Results for the crystallographic directions for the new set of standards in α, β' coordinates are given in Table 1, each position in the table corresponding to a similar position in the triangle in Fig 8b.

PREPARATION AND NATURE OF NEW STANDARDS

A small experimental error occurred

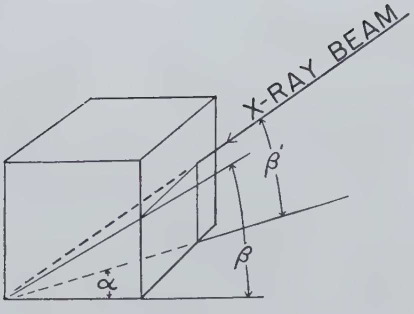
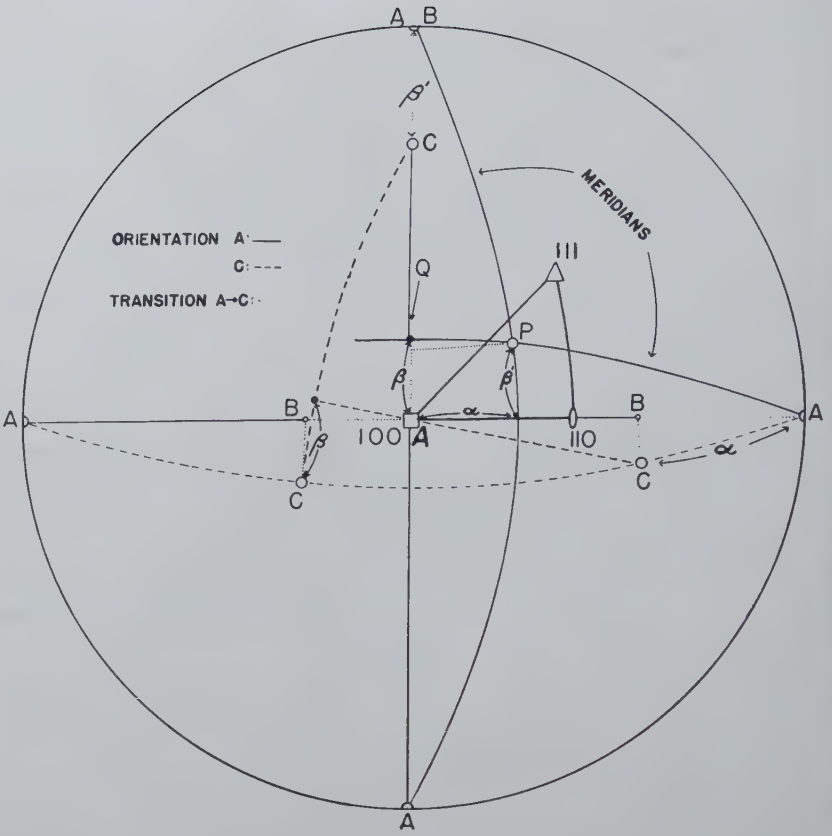


FIG 7—Schematic relationship showing direction of incident X ray beam on a cubic crystal.

(a) Three dimensional drawing showing the angles α, β, and β'.



(b) Stereographic projection for X ray beam in the center of projection and the crystal in orientation C, showing the angles α, β, and β' in relation to orientation A.

in obtaining Lauegrams for crystals oriented as outlined in Table 1; so the actual coverage represented by final standards is somewhat poorer than that indicated in Fig 8b. Nevertheless, the error is generally less than one degree, and this has little effect on the usefulness of the standards. The Laue photographs were taken on Kodak type K X ray film without any intensifying screen, using tungsten radiation at 40 kv (higher voltages introduce additional Laue spots—see later note). The distance between film and sample was 3.2 cm. Three crystals of silicon ferrite each 0.2 mm (0.008 in.) thick were used to obtain 75 patterns for the body-centered cubic lattice. No crystal rotation about an axis perpendicular to the X ray beam exceeded 20°. Exposure times were one hour, and the weaker Laue spots were well recorded. One crystal of aluminum 0.4 mm (0.015 in.) thick was used to obtain all 75 Lauegrams for the face-centered cubic lattice. Exposure times were 15 min. for the aluminum crystal.

Lauegrams obtained on 4½ × 5 in. film and with dark Laue spots on a relatively clear background are called the original positives. From them, marked negatives 3¾ in. × 3¾ in. in size were made as follows: first a correctly marked piece of tracing celluloid was superimposed on the positive and the composite was then printed on contrast process ortho film; secondly, this procedure was repeated using another type of celluloid tracing but the same original positive. In this way 300 negatives, to be used in making final positives on commercial ortho film,* were obtained from the 150 original Lauegrams. Except for the very intense Laue spots, which became enlarged in the reproduction process, final positives made on film were good duplicates of the original Lauegrams.

Sets of standards for preliminary analysis may be mounted between large glass plates in the arrangement partially shown in Fig 9 and 10 for the body-centered (set *B*) and face-centered (set *F*) lattices respectively. Here the positions correspond to those in Fig 8b, and individual patterns have the (010) Laue spots along a vertical line above the central image. The [111] and [001] zones of set *B* and the [101] and [001] zones of set *F* are marked in lightly to show continuity of the pattern with rotation of the crystal and to aid somewhat in the more rapid scanning of the group while locating the pattern which is similar to the one being analyzed.

Standards of the second set for use in the final analysis are kept individually in an index file and are marked so that steps to be taken in the analysis are nearly self-evident. Illustrative examples of such standards are shown in Fig 11 to 16. Miller indices for a zone of Laue spots (the zone axis) are enclosed in brackets and located near the zone; those for a Laue spot (the reflecting plane) are located adjacent to the Laue spot. During the final analysis when the angles δ and ϕ are to be measured, the correct standard is taken from the file and superimposed on the film being analyzed. The identification of zones and Laue spots to use are determined quickly in this way, and zones or Laue spots on films do not have to be marked.

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* The authors recommend final positives on film in preference to positives on paper. The authors also will gladly advise about obtaining these standard patterns on film or paper.

Table 1 . . . The 75 Standards in α, β′ Coordinates

1-1	*4-3*	5-1	7-4½	9-8 9-1	*12-10½* 11-4½	13-8 13-1	15-11½ 15-4½	17-15 17-8 17-1	*19-18* 19-11½ 19-4½	21-15 21-8 21-1	23-18½ 23-11½ 23-4½	25-22 25-15 25-8 25-1	*28-25* 27-18½ 27-11½ 27-4½	29-22 29-15 29-8 29-1	31-25½ 31-18½ 31-11½ 31-4½	*33-28.7* 33-22 33-15 33-8 33-1	35-25½ 35-18½ 35-11½ 35-4½	37-29 37-22 37-15 37-8 37-1	*39-32* 39-25½ 39-18½ 39-11½ 39-4½	41-29 41-22 41-15 41-8 41-1	43-32½ 43-25½ 43-18½ 43-11½ 43-4½	*45-35.3* 45-29 45-22 45-15 45-8 45-1

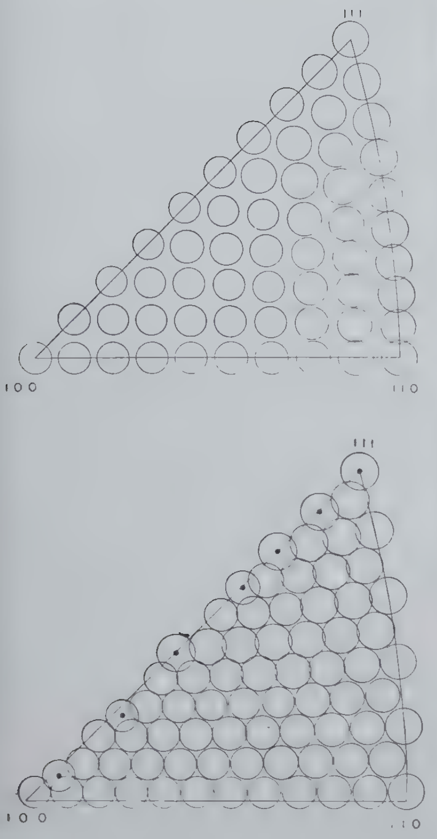


FIG 8—Directions of incident X ray beam in unit triangle form of projection.
(a) (above) The 55 directions used by Majima and Togino in obtaining standard Lauegrams.
(b) (below) The 75 directions used by the authors in a new group of Lauegrams.

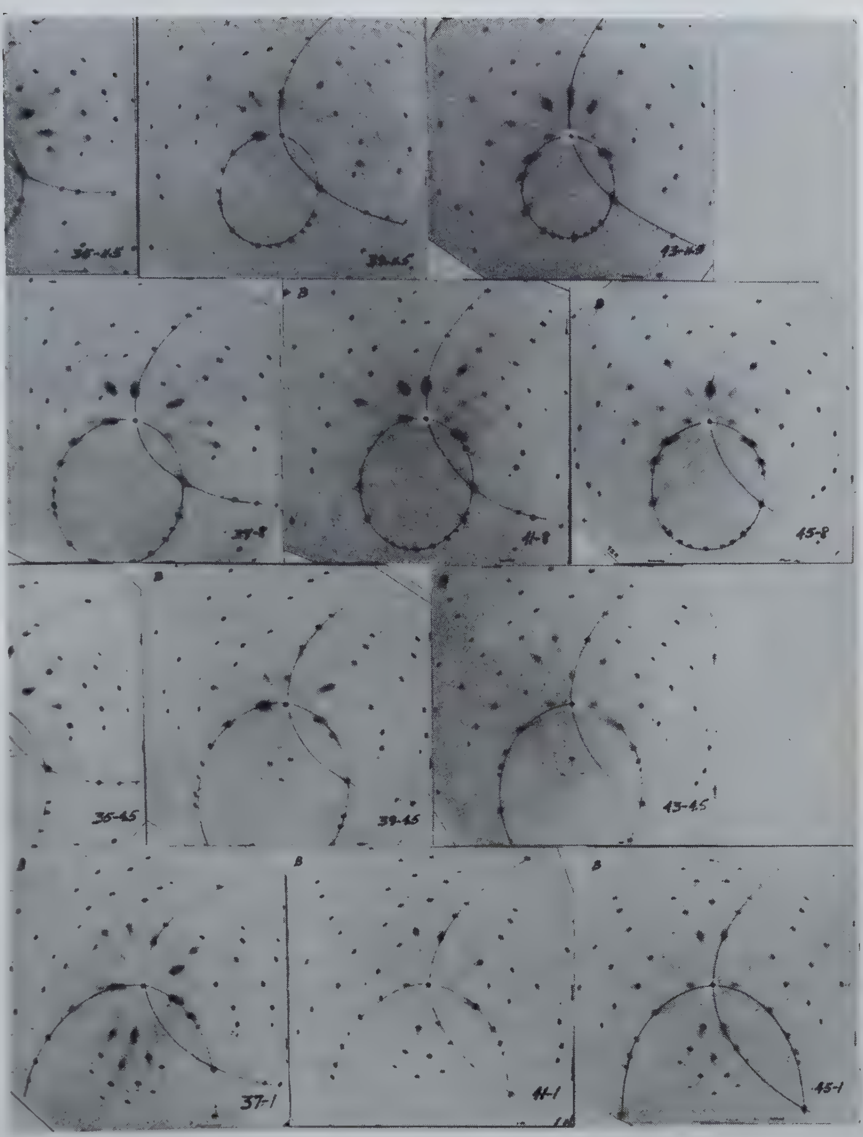


FIG 9—Laue patterns of the body-centered cubic lattice for determining α–β' in a preliminary analysis.

In using standard 1-1 of set *B* shown in Fig 11, it is important to know that the poles of the (031) and $(0\bar{3}1)$ planes fall 143°8′ apart (according to Eq 2). Halfway between them is the pole of the (001) plane. The (010) pole lies on the same meridian 90° from the (001) pole. The [100] direction, the zone axis of the (031) and $(0\bar{3}1)$ planes, lies 90° away, a point easily located with the aid of a Wulff net. The tilt of the (100) plane obtained this way, however, is not accurate; so the pole of the $(\bar{3}01)$ or the pole of the (301) plane should be used to determine the tilt. The [301] direction for example is between the [001] and [100] directions, 18°26′ from the latter. The [111] zone, which is also marked, need not be used except as a check.

The steps of analysis using standard 1-1 of set *F* are the same as those for 1-1 of set *B*. However, the strong <101> zones, which may be used for checking purposes, can be used very conveniently alone if desired to make the complete analysis.

A very simple procedure for carrying out an analysis, which applies to most standards, can be illustrated with standards 29-15 shown in Fig 13 and 14. Plot three poles from the [001] zone including the (010) and $(1\bar{1}0)$ poles if the Laue spots show on the film. Finally, to complete the analysis in terms of cube poles, plot the (001) and (100) poles with the aid of a Wulff net.

In other Laue patterns which do not contain the (010) or the $(1\bar{1}0)$ Laue spot, the procedure of analysis is only slightly modified. Standards of the type illustrated in Fig 15 and 16 (crystals are in the threefold symmetry position) are examples. The poles of the $(\bar{1}30)$ and $(3\bar{1}0)$ planes for the [001] zone, referring to Fig 15, are 126°52′ apart (according to Eq 2); halfway between them is a (110) pole, and this serves to locate the (010) and (100) poles which belong to the [001] zone. The [010] zone need not be used except as a check on the analysis. The {111} poles are sometimes useful in making an analysis; so {111} Laue spots are marked on some standards. Similar in procedure for analysis is the use of the standard shown in Fig 16. The $(\bar{1}20)$ and $(2\bar{1}0)$ poles, in this case, fall 143°8′ apart.

If crystallographic directions other than those marked in the standards are desired, they can be easily located on the stereographic projection from the positions of the cube poles. Therefore, there is no need to index many of the Laue spots. As a matter of fact, it is our opinion that a minimum of indexing of the standards is an aid to more rapid analysis.

It should be pointed out here that there are a number of errors in the standards of Majima and Togino. The errors of indexing can be easily corrected, however, with the aid of equations for crystallographic relations or with standard projections of the poles of planes. Another point con-

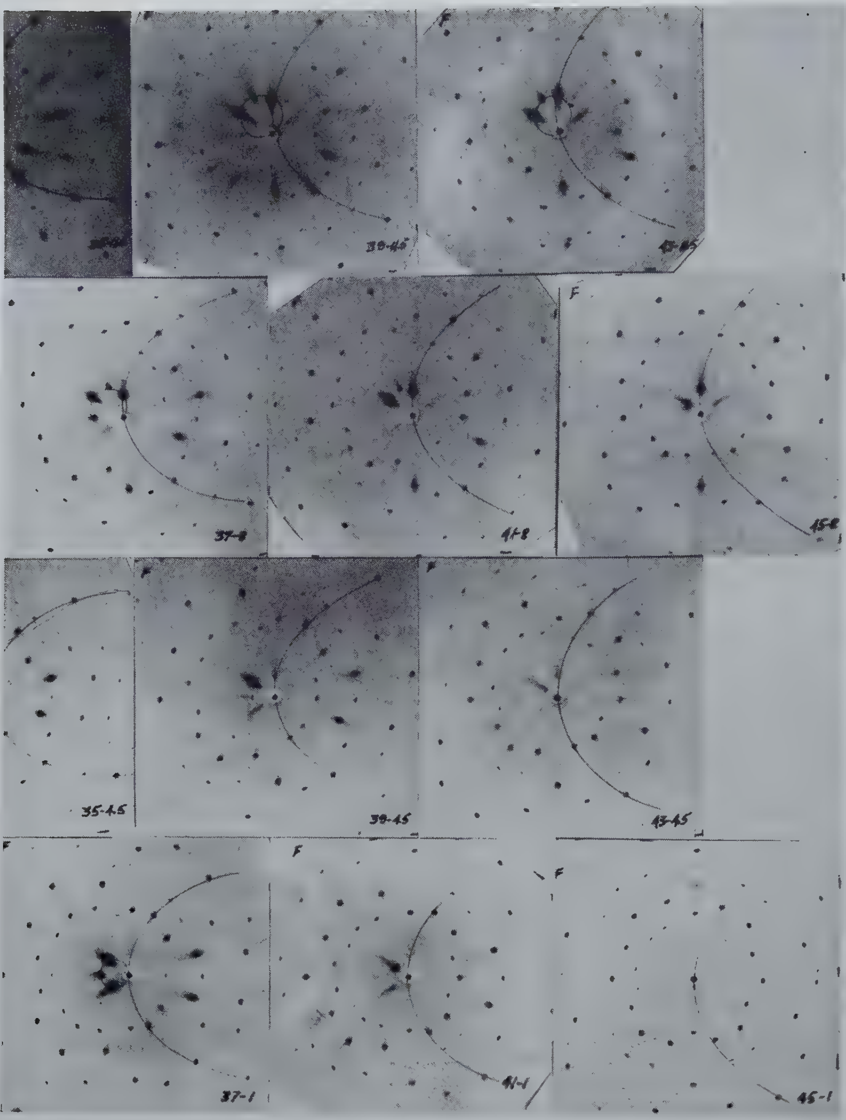


FIG 10—Laue patterns of the face-centered cubic lattice for determining α–β' in a preliminary analysis.

cerns the voltage used on the X ray tube. Referring to standard 45-0 of the body-centered cubic lattice as published by Majima and Togino,[4b] it is observed that {321} Laue spots are present. A voltage of 35 kv was used according to their report; but 40-45 kv peak voltage is needed theoretically to bring out these particular Laue spots. On the other hand, the same Laue spots are absent in our 45-1 standards exposed at 40 kv peak voltage and show only on patterns taken at higher voltages.

Analysis

PRELIMINARY ANALYSIS

A preliminary analysis involves determining which standard Lauegram of the 75 mounted between glass plates resembles most closely the unknown pattern. To obtain agreement some films obviously have to be turned over. They also have to be held in the correct rotational position. When held properly, the [111] zone always falls between 6:00 and 7:30 o'clock except near 45-35.3 where Laue spots of the zone fail to register on the photographic film. The [001] zone lies between 1:30 and 3:00 o'clock except near 0-0 where it fails to register on the film. The [101] zone lies between 9:00 and 12:00 o'clock and fails to show near 45-0.

The [111] and [001] zones for the body-centered cubic lattice often can be correctly recognized at a glance; determining the standard to use for analysis is then an easy matter. When difficulty arises in finding the proper standard, film nets can be used as illustrated in Fig 17 and 18 to help identify the zones. The zone of Laue spots falling on the meridian line of the Leonhardt net in Fig 17 clearly shows a sequence of angular separations of 30° (spots on 20, 50, 80, 110 and 140 latitude lines) thus indicating that the zone is of the <111> form. This same relationship as well as a 135° relationship for a <100> zone is shown even more clearly in Fig 18, the common $(1\bar{1}0)$ Laue spot falling on the base line. Having established the [111] and [001] zones of this pattern, the identification of the Lauegram as one very near 29-15 follows quickly upon comparison with the standards.

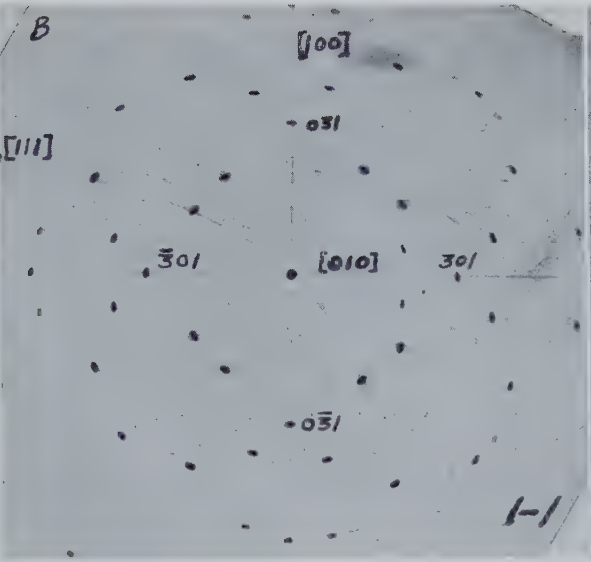
The [101] and [001] zones of the face-centered cubic lattice generally need to be recognized for a rapid identification of the unknown pattern. When difficulty arises, the film nets again can be used as an aid. Although the isogonal net shown in Fig 5 is not constructed for direct application on a [101] zone, it can be used if a rough interpolation only is needed.

FINAL ANALYSIS

In order to plot the orientation of a crystal in stereographic projection, a method for measuring the angles δ and ϕ (see Fig 2 and 3) is required. The δ, ϕ analyzer shown in Fig 19 with δ ranging from zero to 30° and ϕ from zero to 360° fills this need very well; a complete analysis of a film often can be made with it in a time of five minutes or less. A film net as shown in Fig 6 but photographed on glass is mounted in a frame and located over a good source of light. To provide a carrier for the Lauegram, a circular clear piece of film approximately 8 in. in diam is clamped to a metal ring, which is graduated in degrees and is seated in the base concentrically with the film net in such a way that the assembly is free to rotate with the Lauegram while the clear film remains in contact with the surface of the stationary film net. The Lauegram is held against the surface of the clear film with scotch tape, the central spot of the Lauegram coinciding with the center of the film net and the pinprick falling on the single radial line (a reference line) when the scale reads 270. In this position the reference direction *R.D.* should coincide with the zero direction on the scale.

Continuing with the Lauegram used as an illustration in Fig 17 and 18, standard 29-15 from the index file is now laid over the Lauegram on the analyzer. An identified Laue spot is then brought to the reference line of the net. A final position with the (010)

11



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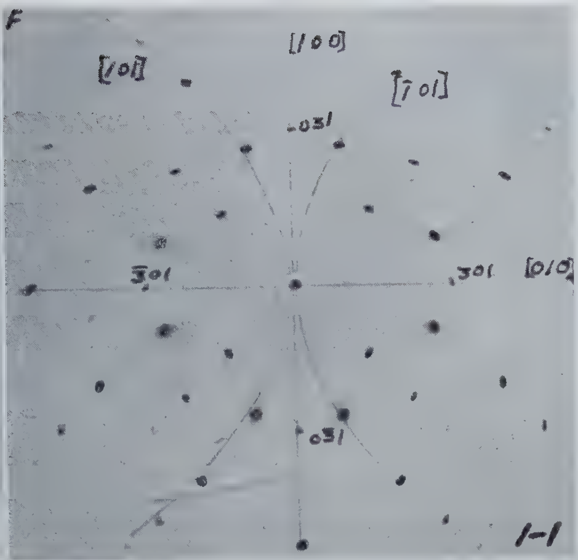
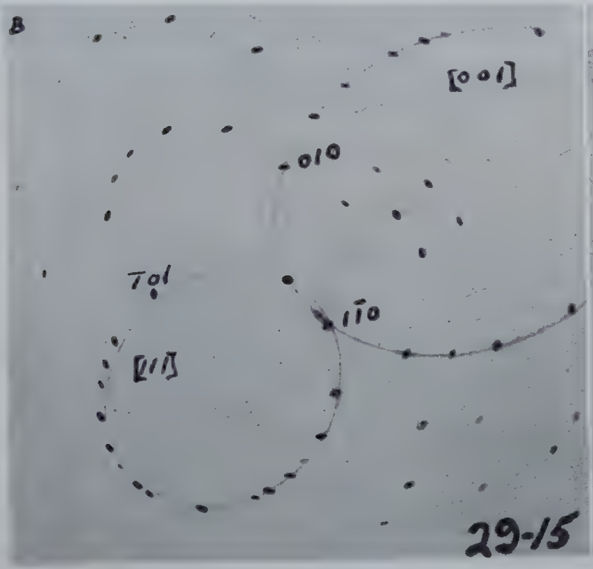


FIG 11—An indexed Lauegram for the body-centered cubic lattice in position 1-1.
FIG 12—An indexed Lauegram for the face-centered cubic lattice in position 1-1.

13



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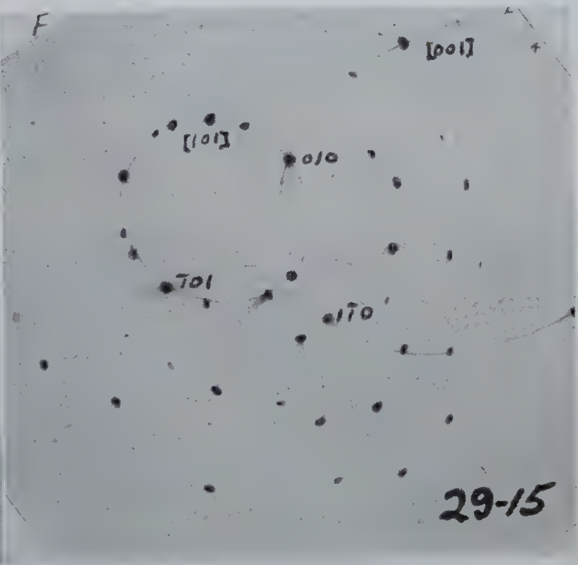
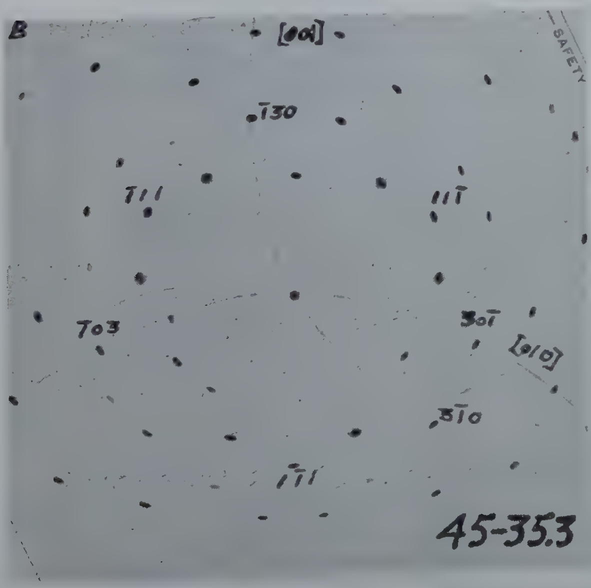


FIG 13—An indexed Lauegram for the body-centered cubic lattice in position 29-15.
FIG 14—An indexed Lauegram for the face-centered cubic lattice in position 29-15.

15



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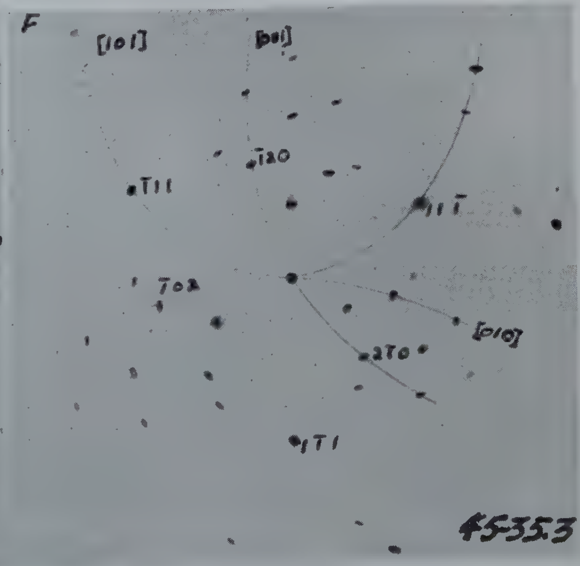

FIG 15—An indexed Lauegram for the body-centered cubic lattice in position 45-35.3.
FIG 16—An indexed Lauegram for the face-centered cubic lattice in position 45-35.3.

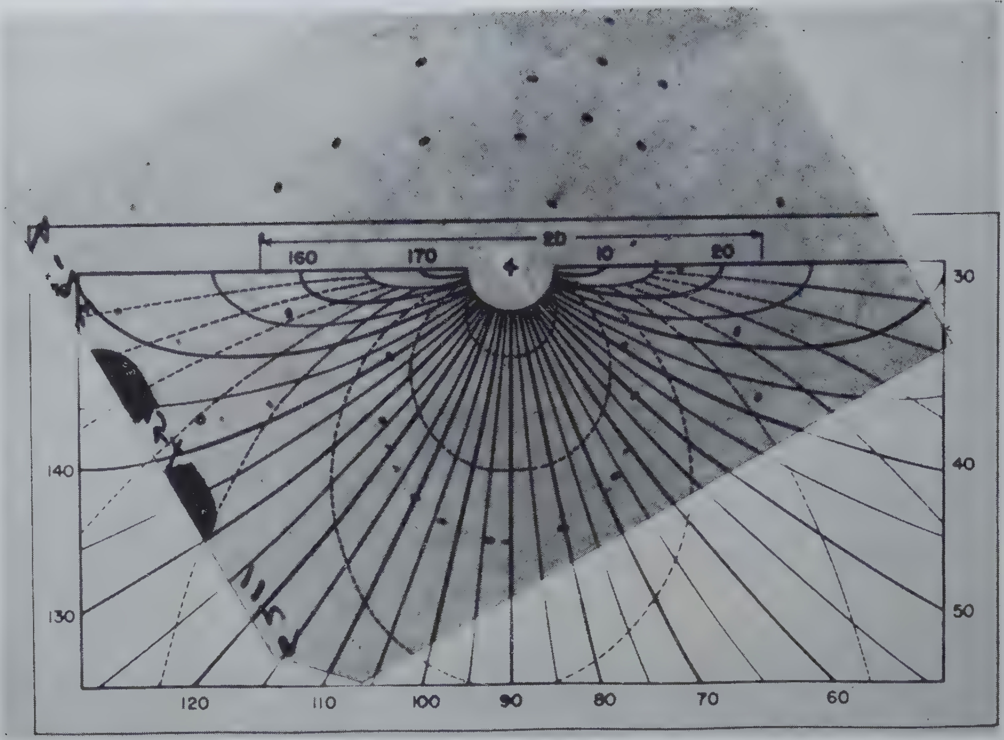

FIG 17—The Leonhardt zero-tilt net superimposed on a zone of Laue spots for measuring angles between planes.

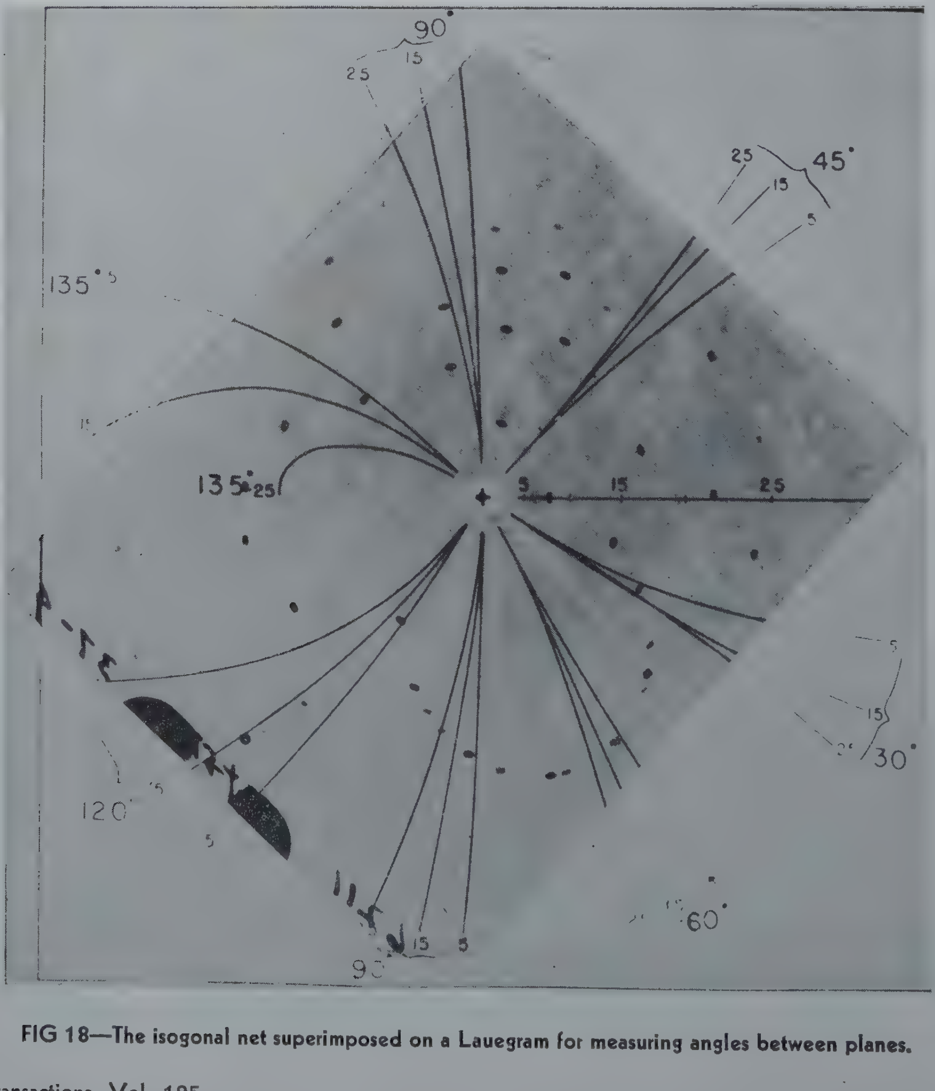

FIG 18—The isogonal net superimposed on a Lauegram for measuring angles between planes.

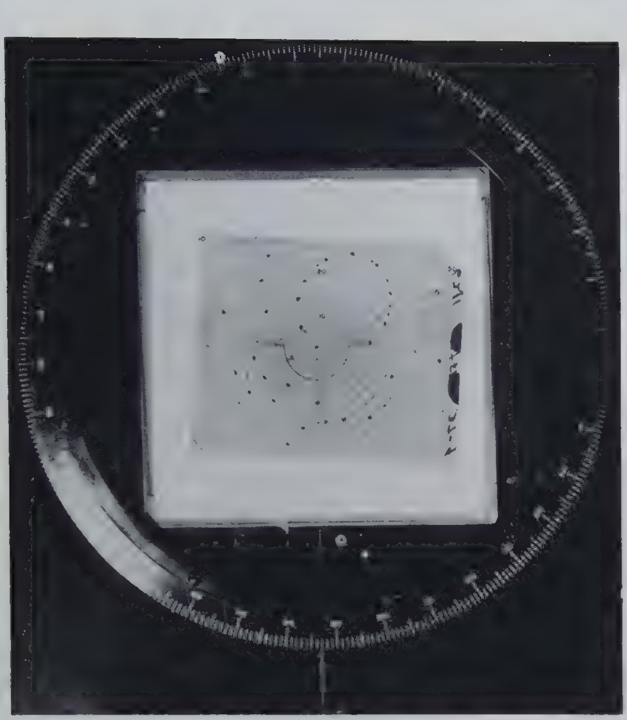

FIG 19—The δ, ϕ analyzer with transmission Laue pattern in position for determining the angles δ and ϕ.

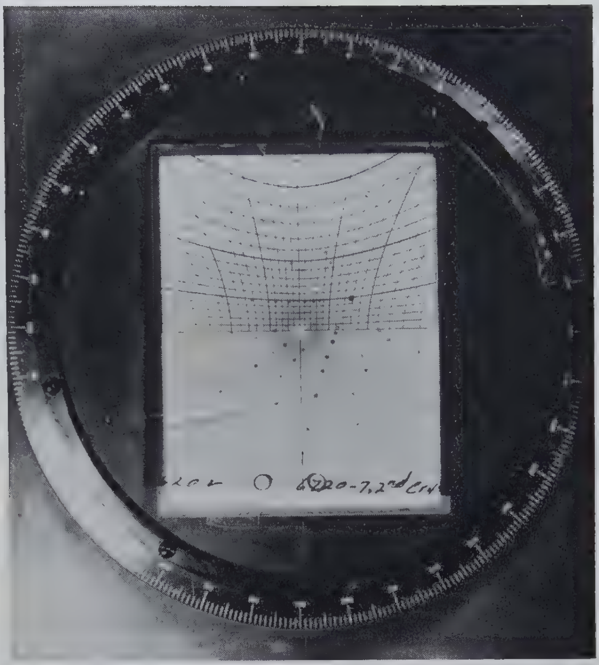

FIG 20—Analyzer employing a Greninger net and showing correct setting for a zone of Laue spots.

Laue spot falling at angles 16° and 187° for δ and ϕ respectively is illustrated in Fig 19. The angles δ and ϕ are essentially polar coordinates and must be so used in plotting the pole of the (010) plane in stereographic projection as was explained in the discussion of Fig 3.* The $(1\bar{1}0)$ and another Laue spot (one with larger δ for greater accuracy) are similarly plotted to determine the [001] zone. With the [001] zone on a meridian line of a Wulff net, the pole of the (100) plane and the pole of the (001) plane—the latter coinciding with the [001] zone axis—can be located directly to complete the analysis for the orientation of the crystal involved.

If a zone axis is to be plotted directly, the angles for doing this are obtained upon placing the zone of Laue spots as parallel as possible to the meridian lines of the net and reading tilt and azimuth from the analyzer.

Final analyses by the present method actually are more difficult to make near some of the common symmetry positions than for other positions. For example, in the face-centered lattice the position 45-0 (twofold symmetrical) is more difficult to analyze than is 29-15 shown in Fig 14. Likewise 0-0 (fourfold symmetrical) for iron is one of the more difficult positions to analyze rapidly.

In the usual back-reflection Laue method, however, symmetry conditions are a great aid to both preliminary and final analyses. Zones are generally more useful than individual Laue spots for plotting the orientation in stereographic projection. A suitable set of marked standards could be made for the more rapid identification of zones in back-reflection Lauegrams.

A convenient method for measuring tilt and azimuth on such patterns is shown in Fig 20. Here a Greninger net comprises part of the analyzer. The Laue pattern is 44-18 in α, β' coordinates. Angle coordinates for a $<111>$ zone, which has been placed parallel to the meridian lines of the net, are a tilt out of the plane of the sample of almost 10° and an azimuthal angle of 191°. These angles can be used with a Wulff net to plot the zone axis in stereographic projection.

Latitude lines on the Greninger net are useful for determining directly angles between planes in the same manner described previously for the zero-tilt Leonhardt net. For example, a $\{112\}$ Laue spot is 30° from the $\{110\}$ spot, the strongest Laue reflection for the $<111>$ zone shown in Fig 20.

Acknowledgment

The amount of work represented by the final marked standard Laue patterns is considerable. The production of the negatives and positives from the original Lauegrams alone is quite a task and the authors are indebted to the Photographic Department of the General Electric Co. for completing this work. The authors are also indebted to Dr. Arthur A. Burr, of Rensselaer Polytechnic Institute, for helpful suggestions in the preparation of the manuscript and to Eugene F. Welter of the Metals Section for assistance in the X ray work.

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* The Wulff net may be marked with ϕ ranging from 0° to 360° in a way to permit direct plotting of the data.

The Vapor Pressures of Zinc and Cadmium over Some of Their Silver Alloys

C. E. BIRCHENALL,* Junior Member, and C. H. CHENG,* Student Member AIME

Introduction

The fundamental problem in the thermodynamics of solid solutions is the determination or calculation of the activities of the components as a function of temperature and composition. Since the theory of metals is not sufficiently developed to allow *a priori* calculation of these quantities, they must be obtained from experiment. C. Wagner[1] has reviewed the literature of this subject for the period before 1940. Although several important and extensive studies have been made in the meantime, the number of systems to which any sort of quantitative information can be assigned is vanishingly small. These data have become of great potential importance in studies of the structure of solid solutions[2] and intermetallic compounds, the nature of the diffusion process,[3,4] and perhaps even the mechanism of mechanical deformation.[5] For these reasons, it seems very worthwhile to extend the experimental data in this field.

Although there is little use in duplicating Wagner's review, attention should be directed to the most recently reported investigations. A brief enumeration of the methods of measurement previously employed also should be valuable. The most direct method is the determination of the partial pressure of the components in the vapor phase in equilibrium with the solution. Both equilibrium and kinetic methods have been tried in metal systems, but almost exclusively on liquid phases. Only in the ease of carbon in iron alloys and in the copper-zinc system have extensive measurements been made which include solid phases.

When one of the components is an element, such as nitrogen or carbon, which forms compounds which are very volatile and stable at normal temperatures and pressures (CH_4, CO_2, CO NH_3), it is frequently possible to equilibrate mixtures containing these (such as CH_4-H_2, CO_2-CO, NH_3-H_2) independently with the elements (C, N) and with the metal (Fe, Ni, etc.). Such measurements have been made for carbon in iron, iron-silicon, and iron-manganese alloys by Smith[6] and in iron and iron-nickel alloys by Toensing.[7] Differences between carbon activity values at the same temperature and carbon content when carbon is introduced from CH_4-H_2 mixtures or CO-CO_2 mixtures indicate that the effects of hydrogen and oxygen in the system are not negligible, and one can only hope that the true value lies somewhere between these sets of results, probably nearer the CH_4-H_2 data since hydrogen is less soluble in iron than oxygen. This is essentially an equilibrium method, although flowing gas is employed.

A kinetic method has been employed which consists of sweeping an inert gas (generally hydrogen) over the alloy at a series of rates, condensing the metal vapor out, and analyzing it. The metal content can be extrapolated to zero flow rate (equilibrium). Wejnarth's[8] work on Cd-Mg and Zn-Mg is a good example of this frequently used method. A variant of this consists of equilibrating a known volume of inert gas with the alloy, sweeping it out quickly, and analyzing for metal. In common with the above method, it has the disadvantage that the "inert" gas is generally somewhat soluble in the alloy. Moreover, the former continuously displaces the system from equilibrium and may give low values if solid diffusion is involved.

The dew point method applied by Hargreaves[9] to the alpha and beta brasses and by Schneider and Stoll[10] to Al-Zn avoids the introduction of another component to the system, but is useful only in systems in which one component is much more volatile than the other. The alloy sealed in one end of an evacuated silica tube is heated to the desired temperature. The temperature of the other end is lowered until droplets of the volatile component condense. When the temperature is raised slightly, the droplets will evaporate. By careful adjustment of temperature, the range between evaporation and condensation can be narrowed appreciably. An independent determination of the vapor pressure of the pure volatile component is necessary to give the partial pressure over the alloy. This is the method employed here to determine the vapor pressures of zinc and cadmium over their silver alloys up to 34 pct in cadmium and 76 pct in zinc. The former involves only the α solid solution, but the latter covers the α, β, γ, and ϵ fields.

In recent determinations, Herbenar, Siebert, and Duffenback[11] used the

Cleveland Meeting, October 1949.
TP 2601 E. Discussion of this paper (2 copies) may be sent to *Transactions* AIME before November 15, 1949. Manuscript received February 9, 1949.

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* Member of Staff and Research Assistant, respectively, Metals Research Laboratory, Carnegie Institute of Technology.

[1] References are at the end of the paper.

absorption of light to determine the concentration of zinc in the vapor phase over copper-zinc alloys. This is probably the method most free from objections arising from disturbance of equilibrium.

To calculate thermodynamic activities with pure metals as the standard states, independent determinations of the vapor pressures of pure components are necessary. For many metals, these have been made by two general types of measurement—the Knudsen method of effusion through an orifice and the method of free vaporization from a surface. The latter usually involves an assumption about the accommodation coefficient of the surface, and insofar as this is in doubt, the data obtained by the method are questionable. Therefore, the Knudsen method is more desirable. A recent experimental triumph is the determination of the vapor pressure of graphite in this manner.[12]

The calculation of thermodynamic activities from vapor pressure data has been described in a recent paper by Birchenall and Mehl[3] for similar data on copper-zinc alloys. The methods are the same here. Another method sometimes employed in the determination of activities involves the measurement of the electromotive force of electrolytic cells. This is only feasible when the electrode reactions can be established beyond reasonable doubt and the cells are reversible in nature.[13]

Experimental

APPARATUS

With the exception of a few minor changes, the apparatus was similar to that of Hargreaves.[9] The alundum furnace tube ($18 \times 1\frac{3}{4}$ in. id) was heated by three main resistance windings, with auxiliary coils to heat the tubes to the observation windows and the end plugs which were inserted to avoid draughts of cold air. The fine adjustment coil was movable in the direction along the tube. A low power cathetometer telescope was used to observe the droplets in the cooler end of the tube. A sketch of the apparatus appears in Fig 1.

TEMPERATURE CONTROL

Since the accuracy and reproducibility of this experiment are dependent chiefly upon the control and measurement of the temperatures at the ends of the fused quartz sample tube, considerable attention has been devoted to these factors. The voltage across each resistance winding could be adjusted independently of the others with "Powerstats." For long heating periods, automatic control equipment was employed. Fluctuations in temperature were quite small after the furnace had been stabilized. It was possible to achieve a controlled temperature difference of 400°C between the ends, considerably more than required in these systems.

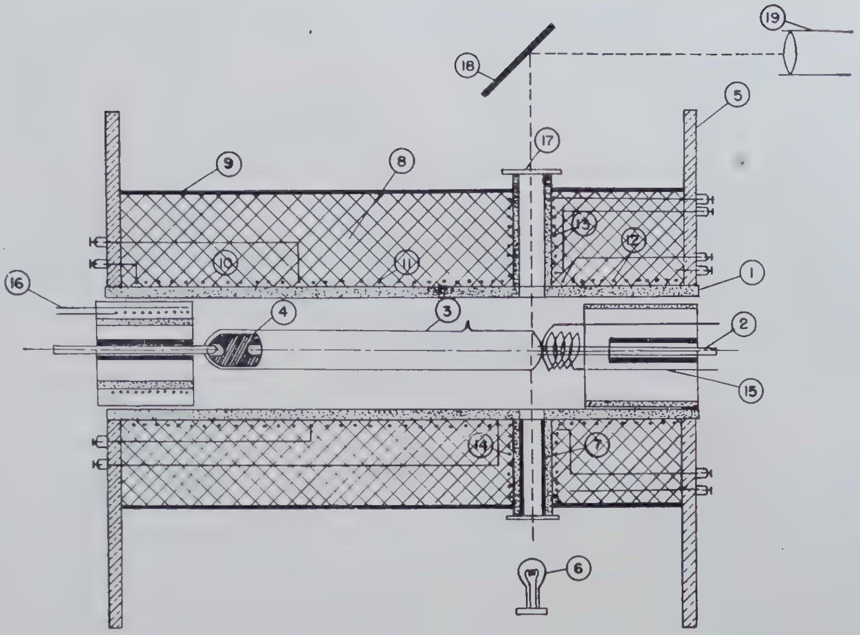

FIG 1—Apparatus for the determination of the vapor pressures of zinc and cadmium over their silver alloys by the dew point method.

1—Alundum furnace tube $18 \times 1\frac{5}{8}$ in. id
2—Quartz thermocouple protection tube
3—Quartz sample chamber
4—Specimen
5—Transite sheet
6—Lamp
7—Reflector
8—Silocel
9—Galvanized steel sheet
10—Coil A, No. 13 kanthal wire, 8 turns per in., 2.8 ohms at 20°C
11—Coil B, No. 13 kanthal wire, 7 turns per in., 2.2 ohms at 20°C
12—Coil C, No. 18 nichrome wire, 4 turns per in., 4.1 ohms at 20°C
13 to 16—Auxiliary coils
17—Glass plate
18—Mirror
19—Telescope

SAMPLE PREPARATION

Silver powder (99.9 pct, lot no. 4203-D, U. S. Metals Refining Co.) was melted by induction heating in a graphite crucible, the inner walls of which were coated with alundum cement. Metallic zinc (99.99 pct) or cadmium (99.95 pct) was added making allowance for vaporization losses. The melt was stirred gently with a fine quartz tube and allowed to stand for about 2 min. before being permitted to freeze in the crucible. After a thin layer of the surface was machined off, the ingot was sealed in an evacuated pyrex capsule and homogenized for 24 hr at 650°C. From this a cylinder was machined about $\frac{5}{8}$ in. high by $\frac{7}{16}$ in. diam and a shallow well drilled in one end to receive the thermocouple protection tube. The samples weighed about 10 g.

PROCEDURE

The specimen was sealed in a transparent quartz tube after flushing several times with dry hydrogen and pumping to a few microns pressure. The tube was inserted in the furnace and heated overnight, the high end stabilized by a controller. The run began by lowering the temperature at the other end slowly until droplets condensed in the field of the telescope. These were reevaporated by raising the temperature. Then the cycle was repeated several times, more slowly, to narrow the range between condensation and evaporation. The differences could be reduced to about 2°C at 10 mm pressure, 3°C at 5 mm pressure, and 4–5°C at 1 mm pressure. The times required to establish these were about 15 min. at 10 mm and 2 hr at 1 mm. The true equilibrium temperature was assumed to lie midway between the condensation and evaporation temperatures.

It was customary to begin with a specimen temperature about midway between the extremes then take the upper and lower points. This prevents overlooking changes which might occur progressively in the alloy to produce errors which would appear systematic if the temperature was always changed in the same direction.

SAMPLING

Samples for chemical analysis were taken from chips removed in shaping the samples before the run, others were machined off after the run. In only one case did these disagree by more than duplicate analyses on the final sample. This indicates that the homogeneity of the cylinders was quite satisfactory. Even if heterogeneity existed on a scale too small to be detected by the sampling method, it would have little effect on the results if the surface was representative. Vapor transport would quickly homogenize the surface where the determination was really made.

In a number of alpha Ag-Zn runs, the droplets formed at the low temperature end were collected and analyzed to determine the validity of the assumption that the condensate consisted of nearly pure zinc. Only the lowest zinc sample showed even a trace of silver, too small to be determined quantitatively. Under the conditions of the analysis, it could not have been as great as 0.1 pct. This was checked by spectrographic analysis which also revealed minute traces of iron, silicon, magnesium, and copper.

Because of its greater volatility, conditions were even more favorable in the cadmium case, and no attempt was made to analyze the droplets.

ERRORS

Although no exact determination of the errors is possible, a reasonable estimate may be given. These are principally of two types—those associated with establishment, measurement, and control of temperatures and those relating to the composition of the sample and droplets. Errors may also be introduced into the calculations due to errors in the vapor pressure data for pure zinc and cadmium, but it is believed that the equations K. K. Kelley[14] employed here are correct to within a few per cent.

Temperatures were measured with chromel-alumel thermocouples standardized against the freezing point of pure aluminum and a Bureau of Standards secondary standard couple. At these low operating temperatures, no contamination or recrystallization of the couples was observed. There was a possibility of error in the failure of the couples to measure the effective temperature of the sample or droplets due to the steep thermal gradient in the furnace. However, care was always taken to see that the specimen covered one thermocouple well, and the droplets formed on the other. A 1°C error in temperature reading produced a relative error of about 1½ pct in the calculations. It seems probable that the error from this source averages less than 5 pct for individual points and less when several points are correlated as below.

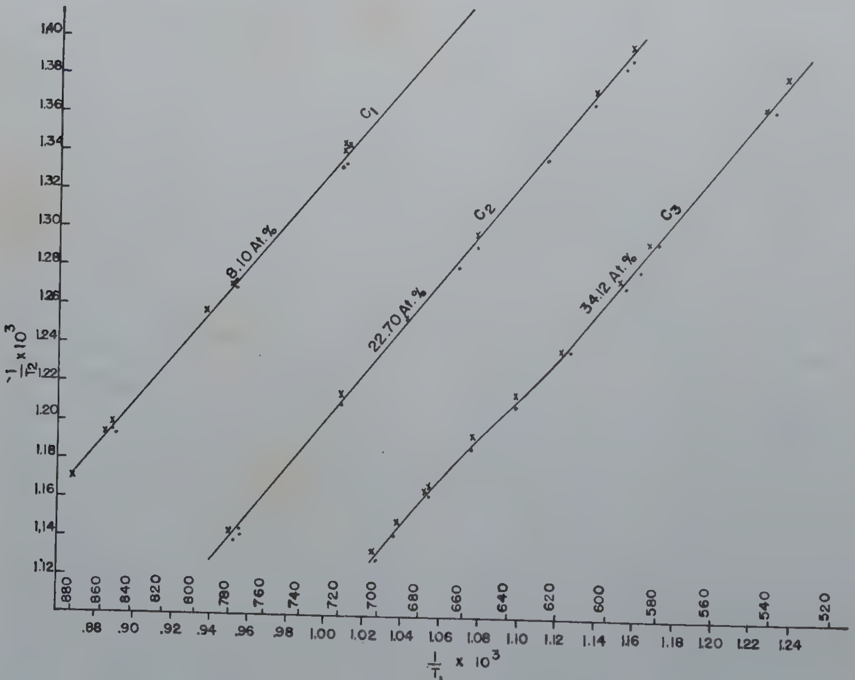

FIG 2—Graph of $1/T_1$ vs. $1/T_2$ for alpha silver-cadmium alloys. T_1 is the temperature of the alloy in equilibrium with cadmium droplets at temperature T_2.

Of the compositional errors, those arising from impurities appear to be negligible. Pure metals were used in melting, and any oxygen introduced would be tied up as ZnO. The latter would make the analyzed Zn content slightly higher than that participating in the process. To minimize this, the surface was cleaned several times before sealing in the quartz tube. The spectrographic analyses indicate that the zinc may have reduced a trace of silicon from the quartz tube.

Heterogeneity of composition may have been present from three sources. Since the specimens were made from castings, some segregation may persist even after fairly long annealing. But the experiment itself is an annealing process, and the samples taken at the end should represent the real composition well. Furthermore, the reproducibility of points was found to be quite good at different stages during the run.

Long heating in a temperature gradient could produce a concentration gradient in the specimen, but the vapor pressure would still approach the true value for the average composition and temperature. It is unlikely that this effect is important here.

Repeated evaporation and condensation might cause considerable differences between the surface and the interior of the sample. In the 20 cm long tube of about 1.3 cm id, it is possible to have 0.01 g of zinc in the vapor phase at 100 mm pressure. This is increased by the necessity of forming droplets of finite size for visual resolution.

There should also be a time dependent hysteresis in the surface layer, as zinc is first evaporated, then condensed. However, by slowing the rate of temperature changes, the gap could be narrowed to the point that the equilibrium value was in little doubt.

Since the analytical deviations average between 1 and 2 pct, the calculated activities may contain errors which aggregate to more than 10 pct for

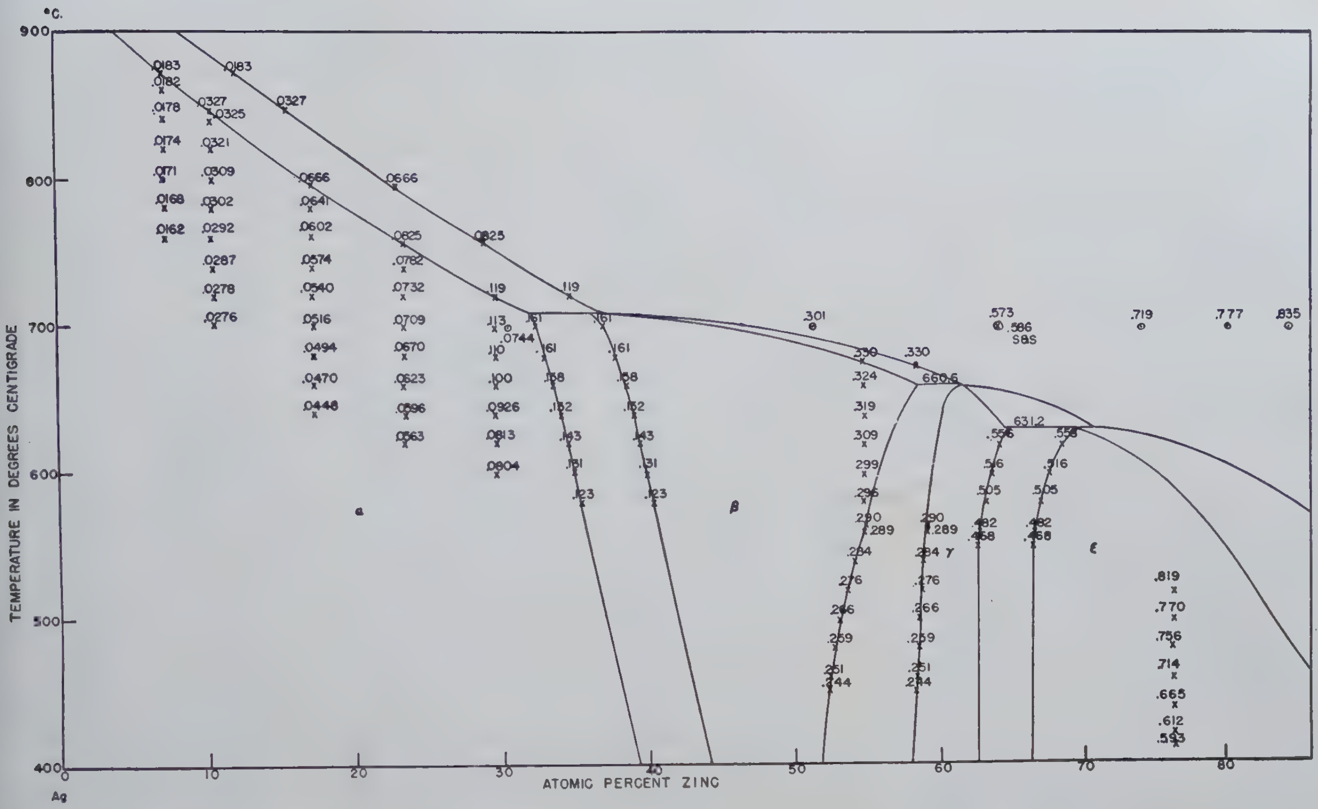

FIG 3—Phase diagram of the silver-zinc system according to Andrews, Davies, Hume-Rothery and Oswin, with the activities of zinc referred to pure liquid zinc as the standard state. The circled points are those of Schneider and Schmid.

single points, but the data taken as a whole should be better than this.

Discussion of Results

The Clapeyron-Clausius equation

$$\frac{\partial \ln p}{\partial T} = \frac{\Delta H}{RT^2} \qquad [1]$$

relates the vapor pressure, p, the absolute temperature, T, the gas constant, R, and the heat of vaporization, ΔH. Over a narrow range of temperature, ΔH will be nearly constant. Then

$$\ln p = -\frac{\Delta H}{RT} + C. \qquad [2]$$

If p_1 is the partial pressure of zinc or cadmium over the alloy at temperature T_1, and p_0 is the vapor pressure of zinc or cadmium over the pure liquid metals at T_2, they will obey

$$\ln p_1 = -\frac{\Delta H_1}{RT} + C_1 \qquad [3]$$

$$\ln p_0 = -\frac{\Delta H_0}{RT} + C_0 \qquad [4]$$

At equilibrium, $p_1 = p_0$, and

$$\frac{\Delta H_1}{R}\left(\frac{1}{T_1}\right) = \frac{\Delta H_0}{R}\left(\frac{1}{T_2}\right) + C_1 - C_0. \qquad [5]$$

Since R, C_1, C_0 are constants and ΔH_1 and ΔH_0 are nearly so, this is nearly a linear equation in $1/T_1$ and $1/T_2$. This is the form in which the experimental data were plotted for correlation. The best smooth curve was drawn through the points, and values were read off at intervals of about 20°C. (As an example, the curves for silver-cadmium are given in Fig 2.) Tables 1 and 2 list the vapor pressures, activities, and activity coefficients for zinc and cadmium calculated from points read from these curves. It is possible by standard methods to calculate the other thermodynamic quantities for these systems as exemplified by the free energy of solution which will be discussed below.

Liquid zinc and cadmium have been chosen as the standard states for the calculation of activities using the vapor pressure equations of K. K. Kelley.[14] Other standard states might have been employed. Particularly in the alpha solid solution ranges, the infinitely dilute solutions offer an attractive state independent of other determinations, but this is hardly suitable for extension to the other phases. Of course it is a simple matter to recalculate these points on the basis of any arbitrary reference state.

Since the activity coefficients for the constituents of two phase alloys depend on the location of the phase boundaries, it was necessary to assume that the diagram for silver-zinc given by Andrews, Davies, Hume-Rothery, and Oswin[15] is correct. This diagram is given in Fig 3 with the activities of zinc marked on it. Any deviations in this diagram will produce errors in the computed values given here. This problem did not arise in silver-cadmium because no two phase alloys were studied.

The few activities reported for solid phases in both systems by Schneider and Schmid[16] are considerably lower than those observed here. This may be a result of allowing too little time for solid diffusion to supply zinc to the vapor. This seems especially likely since their liquid values seem to be quite consistent with the results obtained by extrapolating our data to the solidus line and with our one liquid point on silver-zinc.

It is interesting to examine the alpha solid solutions of the systems Cu-Cd, Ag-Cd, Ag-Zn, and Cu-Zn from the point of view of size factor, electronegativity, and the free energy of solution of liquid zinc or cadmium in silver or copper. According to Weibke and Matthes,[17] the heat of formation is least in the case of Cu-Cd and increases in the order given to Cu-Zn. This is also the order for the free energy decrease given by Biltz.[18] The data of Schneider and Schmid[16] also leave little doubt that in the liquid phase Cu-Cd shows the least decrease in free energy and Cu-Zn the greatest, but Ag-Zn and Ag-Cd are too close to separate. Using the Gibbs-Duhem equation (see ref. 3), it is possible to obtain activities for silver at 25 at. pct zinc or cadmium for 700°C. The partial molar free energy, $\Delta\bar{F}$, of a substance relative to some arbitrary standard state is given by

$$\Delta\bar{F}_i = RT \ln a_i, \qquad [6]$$

and the total free energy change for a binary solution (a measure of its stability) is obtained from this by

$$\Delta F = X_1\Delta\bar{F}_1 + X_2\bar{F}_2, \qquad [7]$$

where the X's are mole fractions. Substituting from above this becomes

$$\Delta F = RT(X_1 \ln a_1 + X_2 \ln a_2). \qquad [8]$$

This is the equation employed in calculating ΔF in Table 3, corresponding to the reaction

Ag or Cu (solid) + Zn or Cd (liquid) → solid solution (25% Zn or Cd)*

From this table, it appears that the stabilities of the Ag-Zn and Ag-Cd solutions are nearly identical with Ag-Cd slightly favored.

The size factors for the four systems are as follows: Cu-Zn, 1.04; Ag-Cd, 1.03; Ag-Zn, 1.08; Cu-Cd, 1.16. Only Cu-Cd exceeds the 1.15 set as the limiting value for extensive solubility. This is confirmed by the observation that Cu-Cd has an alpha solid solution extending to less than 2 at. pct cadmium while the other systems extend to 30 to 40 pct. Thus the repulsion due to large discrepancies in size makes very little free energy available through mixing copper and cadmium. Schneider and Schmid[16] found that liquid solutions of copper and cadmium show positive deviations from ideal behavior while all the other systems above give negative deviations.

The most reasonable measure of electronegativity should be the Fermi energy of the crystalline metals. The differences should give a rough measure of the attraction between the components. Using the values of Slater,[19] the following are obtained (in volts): Cu-Zn, 1.1; Ag-Cd, 0.8; Ag-Zn, 0.4; Cu-Cd, 2.3. Cu-Cd has already been eliminated by the size factor, so the solutions are favored in the decreasing order Cu-Zn, Ag-Cd, Ag-Zn as observed.

Table 1 . . . Vapor Pressure of Silver-Zinc Alloys in the α, β, γ and ϵ Fields

Phase	T°C	P_{Zn}	P°_{Zn}	a	γ
		7.34 At. pct Zn			
α + liq.	872	9.50	520	0.0183	0.249
α	860	8.25	453	0.0182	0.248
α	840	6.55	367	0.0178	0.243
α	820	5.08	292	0.0174	0.237
α	800	3.98	233	0.0171	0.233
α	780	3.05	181	0.0168	0.229
α	760	2.30	142	0.0162	0.221
		10.53 At. pct Zn			
α + liq.	847	13.00	398	0.0327	0.310
α	840	11.93	367	0.0325	0.309
α	820	9.38	292	0.0321	0.305
α	800	7.20	233	0.0309	0.293
α	780	5.46	181	0.0302	0.287
α	760	4.14	142	0.0292	0.277
α	740	3.10	108	0.0287	0.273
α	720	2.28	82.0	0.0278	0.264
α	700	1.67	60.5	0.0276	0.262
		17.33 At. pct Zn			
α + liq.	796	14.70	222	0.0662	0.382
α	780	11.60	181	0.0641	0.370
α	760	8.55	142	0.0602	0.347
α	740	6.20	108	0.0574	0.331
α	720	4.43	82.0	0.0540	0.312
α	700	3.12	60.5	0.0516	0.298
α	680	2.20	44.5	0.0494	0.285
α	660	1.51	32.1	0.0470	0.271
α	640	1.03	23.0	0.0448	0.259
		23.45 At. pct Zn			
α + liq.	757	11.30	137	0.0825	0.352
α	740	8.45	108	0.0782	0.333
α	720	6.00	82.0	0.0732	0.312
α	700	4.29	60.5	0.0709	0.302
α	680	2.98	44.5	0.0670	0.286
α	660	2.00	32.1	0.0623	0.266
α	640	1.37	23.0	0.0596	0.254
α	620	0.90	16.0	0.0563	0.240
		29.65 At. pct Zn			
α	720	9.75	82.0	0.1189	0.401
α	700	6.82	60.5	0.1127	0.380
α	680	4.89	44.5	0.1099	0.371
α	660	3.20	32.1	0.0997	0.336
α	640	2.13	23.0	0.0926	0.312
α	620	1.30	16.0	0.0813	0.274
α	600	0.90	11.2	0.0804	0.271

It is apparent that the size factor and electronegativity correlate quite well in a qualitative way with the observed free energies for these metallic solid solutions. However, it should be emphasized that it is unlikely that constants for the pure metals in themselves give an adequate representation of the state of these atoms in solution. This need not detract from their great usefulness which has been demonstrated in many instances.

The similarity in the deviations from ideal solution laws in the systems Ag-Zn and Ag-Cd to those in Cu-Zn suggests that the same considerations regarding the progression of structures applied earlier to the brasses[2] should also apply here. The progression of structures α, β, γ is, of course, very similar with only moderate shifts of phase boundaries corresponding to the quantitative differences in activities. The great differences in the free energies in the Cu-Cd system are reflected in the very different appearance of its phase diagram.

Short Range Ordering

Our attention has recently been called to an error in ref. 2* where Eq (18) relating the equilibrium constant, K, for the reaction of bonds

$$A\text{-}A + B\text{-}B = 2A\text{-}B \qquad (15)$$

to the fraction, x, of bonds which are B-B at 25 at. pct B has been mistakenly rewritten in Eq (19).

$$K = \frac{(1-4x)^2}{2x(1+2x)} = 4e^{-\Delta F^\circ/RT} \qquad \text{(correct)} \quad (18)$$

$$K = \frac{(1-4x)^2}{2x(1-x)} = 4e^{-\Delta F^\circ/RT} \qquad \text{(incorrect)} \quad (19)$$

ΔF° is the standard free energy for reaction (15).

When the values of x for 25 at. pct zinc in copper are recalculated for 950°C and 700°C, the first approxi-

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* The liquid states of Zn and Cd are used as reference states since they are the stable phases of the pure metals at the temperatures considered. Later in the discussion of short range order one of the assumptions requires the use of a fictitious solid reference state.

* The authors wish to thank Professor C. S. Samis of the Univ. of British Columbia for discovering this error. The equation numbers from the earlier paper will be used in this section in parentheses.

Table 1 . . . (Continued)

Phase	$N(\alpha)$	$N(\beta)$	$T°C$	P	$P°$	a	$\gamma(\alpha)$	$\gamma(\beta)$
37.15 At. pct Zn								
$\alpha + \beta$	0.3235	0.370	700	9.75	60.5	0.161	0.498	0.435
$\alpha + \beta$	0.3295	0.3795	680	7.15	44.5	0.161	0.489	0.425
$\alpha + \beta$	0.3355	0.3860	660	5.08	32.1	0.158	0.471	0.410
$\alpha + \beta$	0.340	0.391	640	3.50	23.0	0.152	0.447	0.388
$\alpha + \beta$	0.345	0.395	620	2.28	16.0	0.143	0.415	0.362
$\alpha + \beta$	0.350	0.400	600	1.47	11.2	0.131	0.374	0.327
$\alpha + \beta$	0.354	0.404	580	0.94	7.63	0.123	0.348	0.304
54.87 At. pct Zn								
(β + liq.)	0.5487		675	13.5	41.0	0.330	0.600	
β	0.5487		660	10.4	32.1	0.324	0.590	
β	0.5487		640	7.33	23.0	0.319	0.581	
β	0.5487		620	4.95	16.0	0.309	0.563	
β	0.5487		600	3.35	11.2	0.299	0.545	
β	0.5487		580	2.26	7.63	0.296	0.539	
$\beta + \gamma$	0.5487	0.5923	564	1.58	5.45	0.290	0.529	0.489
$\beta + \gamma$	0.548	0.592	560	1.47	5.08	0.289	0.527	0.488
$\beta + \gamma$	0.542	0.590	540	0.930	3.28	0.284	0.524	0.481
$\beta + \gamma$	0.536	0.588	520	0.580	2.10	0.276	0.515	0.470
$\beta + \gamma$	0.531	0.587	500	0.349	1.31	0.266	0.501	0.453
$\beta + \gamma$	0.527	0.586	480	0.207	0.810	0.256	0.485	0.437
$\beta + \gamma$	0.525	0.585	460	0.119	0.480	0.248	0.472	0.424
$\beta + \gamma$	0.524	0.584	450	0.089	0.365	0.244	0.465	0.418

Phase	$(N\gamma)$	$N(\epsilon)$	$T°C$	P	$P°$	a	$\gamma(\gamma)$	$\gamma(\epsilon)$
64.29 At. pct Zn								
γ	64.29		620	8.90	16.0	0.556	0.865	
$\gamma + \epsilon$	63.7	67.8	600	5.78	11.2	0.516	0.810	0.761
$\gamma + \epsilon$	63.3	67.1	580	3.85	7.63	0.505	0.798	0.753
$\gamma + \epsilon$	62.8	66.7	560	2.45	5.08	0.482	0.767	0.722
$\gamma + \epsilon$	62.7	66.5	550	1.91	4.08	0.468	0.746	0.703
liquid			700	34.70	60.5	0.573	$\gamma(l)$ 0.891	

Phase	$T°C$	P_{Zn}	$P°_{Zn}$	a	γ
76.45 At. pct Zn					
ϵ	500	0.940	1.31	0.718	0.940
ϵ	480	0.595	0.810	0.735	0.962
ϵ	460	0.335	0.480	0.698	0.913
ϵ	440	0.181	0.273	0.662	0.867
ϵ	420	0.093	0.153	0.608	0.795
ϵ	410	0.067	0.113	0.593	0.776

Table 2 . . . Vapor Pressure of Alpha Silver-Cadmium Alloys

$T°C$	Pmm. Hg	$P°$	α	γ
8.10 At. pct Cd				
880	63.0	2100	0.0300	0.370
860	50.0	1790	0.0279	0.344
840	40.2	1500	0.0268	0.313
820	31.7	1230	0.0258	0.319
800	24.8	1025	0.0242	0.299
780	19.0	850	0.0224	0.277
760	14.6	695	0.0211	0.260
740	11.1	565	0.0196	0.242
720	8.20	445	0.0184	0.227
700	6.00	342	0.0175	0.216
680	4.36	269	0.0162	0.200
660	3.15	203	0.0155	0.191
22.70 At. pct Cd				
800	117	1025	0.1141	0.503
780	90.5	850	0.1064	0.469
760	70.0	695	0.1007	0.444
740	52.5	565	0.0929	0.409
720	39.1	445	0.0876	0.386
700	29.0	342	0.0848	0.374
680	21.0	269	0.0781	0.344
660	15.0	203	0.0739	0.321
640	10.5	155	0.0677	0.298
620	7.15	115	0.0622	0.274
600	4.82	85.0	0.0567	0.250
580	3.22	62.5	0.0515	0.227
34.12 At. pct Cd				
720	130	445	0.292	0.856
700	96.0	342	0.281	0.823
680	71.5	269	0.266	0.780
660	52.8	203	0.260	0.762
640	40.2	155	0.259	0.759
620	29.8	115	0.259	0.759
600	20.1	85.0	0.236	0.694
580	13.5	62.5	0.216	0.633
560	8.80	43.7	0.201	0.590
540	5.60	30.4	0.184	0.539
520	3.48	20.8	0.167	0.489

P = pressure of zinc or cadmium in mm. Hg over alloy.
$P°$ = pressure of zinc or cadmium over pure liquid metal at the same temperature.
a = P/P°, the activity of zinc or cadmium.
γ = a/N, the activity coefficient.
N = mole fraction of zinc or cadmium.

mations give $\frac{1}{20.4} = (0.0490)$ and $\frac{1}{23.0} = (0.0435)$ respectively. These results are in good agreement with those of Guttman[20] calculated from the equation of Takagi,[21]

$$\frac{v_{AA} + v_{BB} - 2v_{AB}}{kT} = \ln \frac{(c - x)^2}{x(1 - 2c + x)} \qquad [9]$$

x has the same meaning as above, c is the atomic fraction of component B in the solution, and $\Delta F° = N\,(2v_{AB} - v_{AA} - v_{BB})$ where N is Avogadro's number. Eq 9 may then be rewritten in exponential form

$$e^{-\Delta F°/RT} = \frac{(c - x)^2}{x(1 - 2c + x)} \qquad [9a]$$

Inserting $c = 1/4$, this reduces immediately to Eq (18).

Repeating the procedure used in ref. 2 for $c = 1/3$ and $c = 1/2$ and substituting these values in Eq 9a, again gave two sets of identical equations. For

$$c = \frac{1}{3},\ e^{-\Delta F°/RT} = \frac{(1 - 3x)^2}{3x(1 + 3x)}$$

$$c = \frac{1}{2},\ e^{-\Delta F°/RT} = \frac{(1 - 2x)^2}{4x^2}$$

It therefore appears that the methods of Birchenall and Takagi are equivalent for the treatment of short range order.

For calculations of short range order, the pure solid materials make the most convenient reference states. For zinc and cadmium, this involves an extrapolation of the vapor pressure equations for the solid beyond the melting point. In the case of zinc, the extrapolation is probably fairly good but cadmium is rather uncertain. Nevertheless this has been done here in determining the activity coefficients referred to the solid, approximate bond energies, free energies for reaction (15), and the short range order for the systems Cu-Zn, Ag-Zn, and Ag-Cd for 700°C and 25 at. pct Zn or Cd. In an ideal solution x, the fraction of Zn-Zn or Cd-Cd bonds would be 0.0625 ($1/16$), but in all three cases it is less than this. The tabulation follows (Table 4).

Using Darken's[22] correlation of the activity coefficients for solid silver-gold solutions and Eq 9, the x's have been calculated for mol fractions 0.25, 0.50, and 0.75 at 700°C and several temperatures along the solidus line. Even in this completely isomorphous system and so near the melting points the ordering is seen to be appreciable.

The scatter of data did not permit estimating the dependence of $\Delta F°$ on temperature for reaction (15) for silver and gold. Consequently, the mol fractions and x's apply to either component, the deviations showing up most markedly in the value of x for the dilute component (Table 5).

Summary

The vapor pressures of cadmium in equilibrium with three alloys in the alpha solid solution field in silver have been measured over a temperature range of about 200°C. Similar measure-

Table 3 . . . Free Energies of Solution at 25 At. Pct Zinc or Cadmium at 700°C for Ag-Zn, Ag-Cd, and Cu-Zn

System	a_1	a_2	log a_1	log a_2	ΔF
Ag-Zn	0.718	0.0788	−0.144	−1.1035	−1710
Ag-Cd	0.642	0.1060	−0.193	−0.975	−1730
Cu-Zn	0.611	0.0470	−0.214	−1.328	−2190

Note: Subscript (1) refers to Ag and Cu, (2) to Zn and Cd.

Table 4 . . . Short Range Order and Bond Energies for Cu-Zn, Ag-Zn, Ag-Cd at 700°C and 25 At. Pct Zn or Cd

System	(Stabilization Energy of $A - B$ over $B - B$)	ΔF°	$X_{\text{Zn or Cd}}$
Cu-Zn	B = Cu 264 cal per mol of bonds Zn 852	−1116 cal per mol of bonds	0.0435
Ag-Zn	Ag 54.5 Zn 644	− 699	0.0503
Ag-Cd	Ag 202 Cd 536	− 738	0.0492

Table 5 . . . Short Range Order in Silver-Gold Alloys

Atom Fraction	x (ideal)	x (calculated)			
		700°C	990°C	1015°C	1040°C
0.25	0.0625	0.0532	0.0553		
0.50	0.250	0.233	0.237	0.237	
0.75	0.563	0.553	0.555	0.555	0.555

ments have been made on nine alloys of silver and zinc from 8 to 76 at. pct zinc and over a somewhat narrower temperature range. These alloys include the α, β, γ, and ϵ fields, as well as yielding activities along several of the liquidus lines.

The thermodynamic activities and activity coefficients have been calculated for the volatile component in each case. In the silver-zinc system, the activity coefficients of two phase alloys are based on the phase diagram of Andrews, Davies, Hume-Rothery, and Oswin.

At 700°C and 25 at. pct, the free energies of solution for liquid zinc and cadmium in solid silver have been computed for comparison with those of copper-zinc. The free energies of these systems have been compared with each other and copper-cadmium with respect to size factor, electronegativities and the progression of stable phases in the phase diagram.

The methods for calculating short range order developed by Birchenall and Takagi have been shown to lead to identical results. Application has been made to the alpha solid solutions of silver-zinc, copper-zinc, and silver-cadmium at 700°C and 25 at. pct zinc or cadmium and to the silver-gold system. These calculations indicate a greater proportion of A-B bonds than would be present in ideal solution.

Acknowledgments

This work was done on a contract between the Office of Naval Research and the Metals Research Laboratory of Carnegie Institute of Technology. The chemical analyses were performed by Martha M. Helzel. Many of the runs were carried out by Wilbert Walker, and a few of the alloys were melted by William J. Reichenecker. Their assistance is gratefully acknowledged.

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Sintering Characteristics of Minus Sixty-five and Twenty Mesh Magnetite

ALAN STANLEY,* Junior Member AIME, and JOSEPH C. MEAD

Introduction

The MacIntyre Development of the National Lead Co. is located at Tahawus, N. Y. The operations involve the mining and concentrating of a titaniferous iron ore to produce an ilmenite concentrate and a magnetite concentrate.

Construction of the MacIntyre plant was commenced during the summer of 1941, when world conditions threatened to cut off the supply of Indian ilmenite. An open pit mining operation was developed and the crushing and milling equipment put in operation in July 1942. A general description of the operation was given in the Adirondack Issue of *Mining and Metallurgy* for November 1943. The metallurgy of the mill operation was described by Mr. Frank R. Milliken,* Plant Manager, National Lead Company, MacIntyre Development, and presented at the AIME New York Meeting, February 1948.

The magnetite concentrate produced in the milling operation was too fine (minus 20 mesh) to be used directly in iron blast furnace operation, and most of the magnetite had to be stockpiled in 1942 and 1943.

In 1943, the Defense Plant Corp. built a Greenawalt sintering plant at Tahawus, N. Y., to put the magnetite concentrate in a more suitable form for use in the iron blast furnace.

The Greenawalt sintering plant consists of three 10 by 25 ft sintering pans designed to produce 1800 gross tons of sinter per 24 hr. The vacuum to each pan is produced by two Greenawalt fans in series, pulling approximately 30,000 cu ft of air per minute at 50 in. water gauge vacuum. The plant started operation in August 1944. The present plant production averages 25 tons per operating pan hour (approximately 224 lb per operating hour per square foot of grate area) of plus 1 in. sinter.

Raw feed to the plant consists of 61 pct magnetite, 4 pct anthracite coal culm, and 35 pct minus 1 in. return fines which are conveyed to a pug mill where the materials are mixed thoroughly and water added to give the mixture 5.5 to 6 pct moisture. The mixed prepared feed is conveyed to two 4 by 10 ft vibrating screens where the minus 1 in. plus ⅝ in. return fines are screened out and discharged into a surge bin for use as a hearth layer. The minus ⅝ in. prepared feed is discharged into another surge bin for use as prepared feed.

A charge car, electrically operated, having a capacity of one charge of prepared feed and several charges of hearth layer, lays a thin layer of plus ⅝ in. return fines and 9½ in. depth of prepared feed into the pans. A fluffing roll and a vibrator on the car fluffs and spreads the prepared feed into the pans.

An ignition car, electrically operated, ignites the top of the bed with a 30 sec flash burn. The 9½ in. bed sinters in approximately 13 min. Dumping the pan, and recharging and igniting the bed requires 2 min.

To improve the quality of the ilmenite concentrate produced in the mill and to reduce the amount of titanium dioxide lost in the mill tailings and in the magnetite product, extensive research work and pilot plant operations have been done on grinding the crude ore to minus 65 mesh size (rather than to minus 20 mesh) and concentrating it by a combination of magnetic separation (for magnetite recovery) and flotation (for ilmenite recovery). These tests have proved successful in increasing ilmenite recovery and grade.

With the development of the ilmenite flotation process to a stage where a full scale flotation plant was in the design stage, the problem arose of handling the 65 mesh magnetite concentrate that would be produced. In order to study and solve the problems of handling and sintering the 65 mesh magnetite in the sinter plant, a pilot sinter plant was secured from John E. Greenawalt.

The effect of using 65 mesh magnetite in the sintering operations was then studied on the 2.4 sq ft test pan, operating under conditions as similar to the large plant as could be set up in the laboratory.

A series of tests were run in the test pan on present sinter plant feed that had been mixed in the plant pug mill. An average production and an average quality of sinter produced in this series

San Francisco Meeting, February 1949.

TP 2597 BC. Discussion of this paper (2 copies) may be sent to *Transactions* AIME before Aug. 1, 1949. Manuscript received Nov. 27, 1948.

* General Engineer and Sinter Plant Superintendent, respectively, MacIntyre Development, Titanium Division, National Lead Co., Tahawus, N. Y.

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* Frank R. Milliken: Metallurgy at National Lead Company. TP 2355, AIME *Mining Tech.* (May 1948).

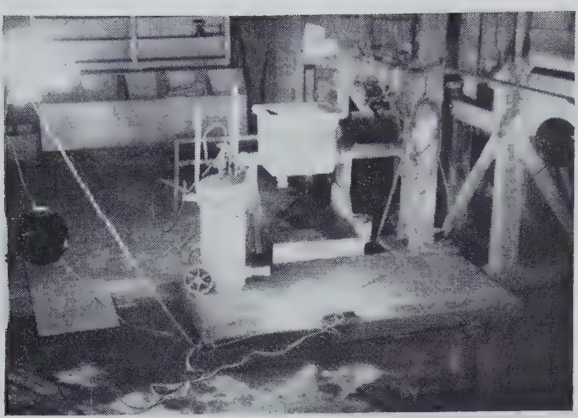


FIG 1—Sinter pan and kerosene burner.

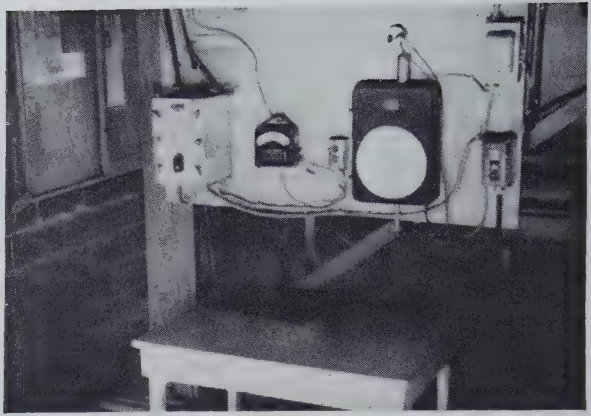


FIG 2—Instrument panel.

of tests was established as a "Standard" (8.8 lb per min) to compare the sintering of minus 20 mesh magnetite with that of minus 65 mesh magnetite.

The high points on each series of tests were repeated twice as a check on the original, to be sure that some unknown variable did not cause the change in the sintering performance. A test run of 10 pans was made on the mixture found to give the best production and quality.

Procedure

The pilot sinter plant included a sintering pan 20¾ by 16¾ in. and 9 in. deep, an ignition hood, a pressure kerosene burner for ignition, a dust collector, and a 400 cfm vacuum fan rated at 54 in. of water (see Fig 1 and 2 for instrument panel, sinter pan and kerosene burner).

Vacuum was recorded on a Bristol meter, and exhaust temperatures were read from an electrical pyrometer.

Magnetite of 65 mesh was prepared by grinding stockpile (20 mesh) magnetite, to the desired size, in a batch ball mill, and filtering in a laboratory pressure filter, Table 1.

The moisture obtained by this method of filtering was too high for pilot plant use (averaging about 9 pct) and it was necessary to dry a portion of the magnetite prior to its use in the sintering operation.

For the final "production run" tests, rod mill screen undersize (20 mesh) was ground to 65 mesh, and the magnetite was concentrated in a laboratory Crockett.

Coal was obtained from the sinter plant stockpile, and when necessary, ground in a ball mill to the desired size. After several tests had to be discarded because of poor results caused by coal with high ash content, all coal samples were submitted to the laboratory for ash determination. In order to eliminate one variable, only coal with a 10 pct or less ash content was used in the test work.

Sawdust, when needed, was obtained from local mills; limestone and slaked lime were obtained from the Chazy Lime and Stone Co.

The return fines, unless otherwise noted, were obtained by crushing plus 1 in. sinter, produced in the pilot plant, in a jaw crusher and rolls and screening it to the desired size on a laboratory vibrating screen.

Screens for screening the sinter were made locally, as were sample racks and storage bins for the raw materials. A ¼ yard gasoline concrete mixer was connected to an electric drive and used for the mixing of all batches. Balances and a hot plate were provided for moisture determinations, and an electric clock with a sweep second hand was used for timing the tests.

A group of five persons was found to be the most efficient, as continuous test work required constant work by one man grinding magnetite and coal, one man preparing sized return fines, two men preparing batches for burning, and one man completing the necessary calculations and paper work. The last three men were required when the batches were burned.

Table 1 . . . Screen Analysis of Magnetite Products

	+28	+35	+48	+65	+100	+150	+200	−200
20 mesh	8.3	16.4	19.5	14.8	12.1	9.2	5.2	14.5
65 mesh				3.0	11.2	19.8	15.8	50.2

Note: All references to 65 and 20 mesh magnetite refer to minus 65 and minus 20 mesh material.

Each batch mixed contained enough material for two tests. The charge was calculated in advance. A weighted mixture of dry and filtered magnetite was added to the mixer. This material was then mixed for 5 min and a moisture determination made. Using this moisture it was possible to calculate the exact amount of magnetite that had to be removed or added to the mixer to leave the required weight of magnetite at 5 pct moisture.

Then the remaining materials were added dry, and the necessary amount of water to provide the required moisture was sprinkled, with a sprinkling can, into the revolving mixer. When trouble with pellet formation occurred, the procedure was changed so that the necessary water was added to the return fines, and then the wet returns were added to the mixer. The latter method was more satisfactory.

Each batch, unless otherwise noted, was mixed for 5 min again.

A 15 lb bedding charge (minus 1 in. plus ½ in. sinter) was added to the pan for each burn. This gave a bedding depth of 1 in. Then the sinter pan was filled, with a weighed amount of mix, and dumped by hand through a 1 in. screen. The bed then was made level by scraping off the excess material. The weight of the mix in the pan was obtained by difference. The ignition hood was lowered into place over the pan, and the pressure kerosene burner was ignited, as the vacuum was turned on, and ignition took place for 30 sec. The hood was then raised. A record was made of the exhaust temperature as the burning proceeded and vacuum was recorded continuously with the Bristol meter. When the bedding on the grate bars glowed red (viewed by means of a mirror mounted to make the grates visible through a hole covered with glass in the bottom of the pan) the vacuum was turned off and the sintered charge was dumped by inverting the pan.

The results of the first pan of the mix were usually poor because a cold pan did not approximate actual operating conditions. The second, or hot pan, gave results that could be compared more directly with sinter plant practice.

After each pan was burned, the material was passed over a 1 in. Ty-Rock screen three times. The material remaining as plus 1 in. was "sinter." The minus 1 in. material was passed over a ½ in. Ty-Rock screen twice, and the plus ½ in. material was "bedding." The remainder of the material (minus ½ in.) was "return fines" (note: in all references to return fines in this paper, the bedding is excluded).

In order to have a strength comparison of the sinter formed by various mixes, a "quality" figure was initiated. This figure was determined by screening the cold (the plus 1 in. sinter was allowed to air cool overnight) sinter in the same manner as above. The weights of the plus 1 in. and the plus ½ in. were combined, and this total weight was divided by the weight of the hot 1 in. sinter to obtain relative "quality."

Cycle time was figured by adding 2½ min to the burning time to allow for dumping and recharging the pan. Theoretical production in pounds per minute was determined by dividing the pounds of plus 1 in. sinter by the cycle time. In quite a few tests the amount of bedding and return fines produced was not sufficient for repeat operations. Because of this some of the plus 1 in. sinter had to be taken to provide the amounts of these materials needed. Actual production was then the weight of plus 1 in. sinter remaining after bedding and return fines requirements were met, divided by cycle time.

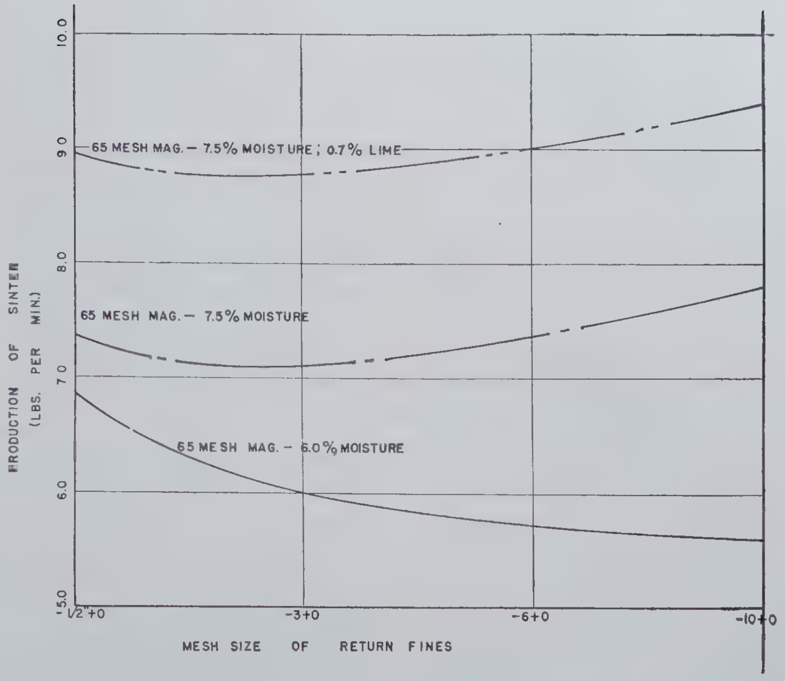


FIG 3—Results of return fines series.

Discussion of Factors Investigated

The following factors were investigated during the test period:

(1) return fines, (2) coal, (3) moisture, (4) slaked lime and miscellaneous reagents, (5) oxidation, (6) limestone, (7) sawdust, (8) pellets, (9) "dust," (10) mixing time, and (11) production runs.

RETURN FINES

As far as is known, there is no sinter plant in the United States that employs sized return fines as a necessary metallurgical requirement for its operation. Mr. Worm Lund, on a visit to MacIntyre from Sweden, suggested the possibility of sized return fines in the sintering of 65 mesh magnetite.

In the test plant at 20 mesh, return fines were not necessary for the production of good sinter. In many cases the return fines were detrimental to test plant production. This is especially true when the returns consisted mainly of raw magnetite, coal, and dust from the dust collectors.

For sintering 65 mesh and finer magnetite, return fines are absolutely necessary for sinter production. If efficient operation and economical sinter are to be had, it is necessary to have these return fines fall within definite size ranges. The secret of sintering fine concentrates is to obtain proper porosity in the "bed" so that air can be uniformly distributed throughout and burning can progress.

A series of tests were run using minus ½ in. plus 3; minus 3 plus 6, minus 6 plus 10, minus 10 plus 20, minus 10 plus 35, and minus 20 plus 35 mesh return fines. The best results were obtained from the minus 10 plus 35 mesh tests. Because of the difficulty that would be incurred by screening to these close sizes, a series was run using minus ½ in. plus 0, minus 3 plus 0, minus 6 plus 0, and minus 10 plus 0. The results of this series can be seen in Fig 3. These tests were run with 4.0 pct coal and 25 pct return fines.

As can be seen (Fig 3) the best results were obtained using minus 10 mesh plus 0 return fines. The best percentage of this size return fines was determined by a series of tests (Fig 4). The results were satisfactory only as long as the minus 35 mesh portion did not exceed 25 pct of the total return fines weight. (This then would give a calculated minus 10 plus 35 mesh return fines figure of 18.75 pct of the total weight of the charge.)

A study of the results obtained using various amounts and sizes of return fines indicated:

1. Proper porosity is essential in producing sinter economically:
    - *a.* Too much porosity causes too rapid burning and results in poor sinter.
    - *b.* Insufficient porosity increases the burning time beyond economic limits.
2. Excess returns weaken the sinter and cause a drop in production.
3. By proper control of the size and amount of returns it should be possible to sinter magnetite finer than 65 mesh.
4. More efficient sinter plant operation could be obtained if the dust from the dust collectors and the finer portion of the returns were returned to the original source of sinter plant feed (for example: to magnetite filter).

A 20 mesh magnetite will sinter without the addition of return fines, providing adequate bedding is supplied, but blast furnace operations object to fines; this requires that the fine material be screened out and recirculated.

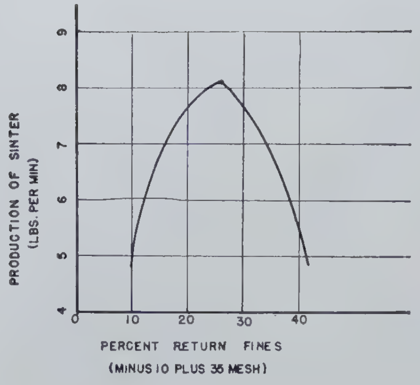


FIG 4—Results of various percentages of minus 10 plus 35 mesh returns.

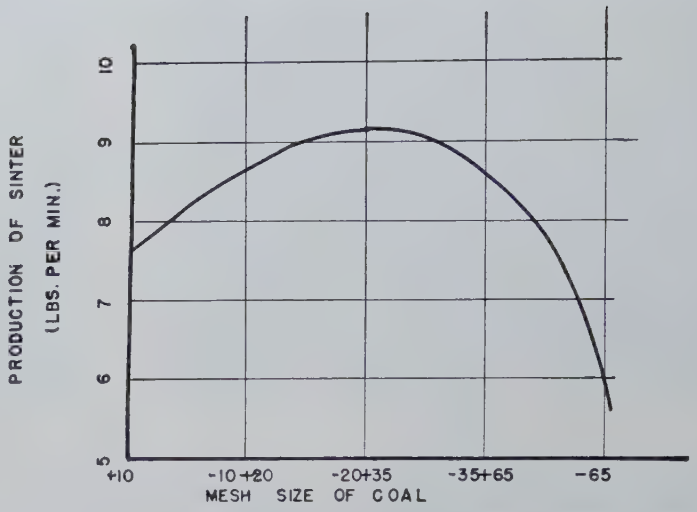


FIG 6—Effect of various mesh sizes of coal on sinter production using 65 mesh magnetite.

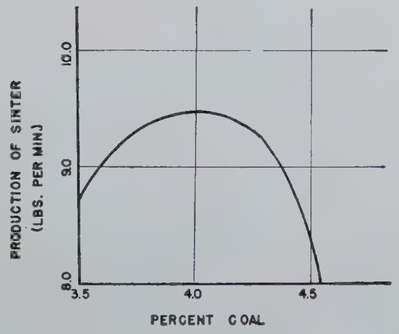


FIG 5—Effect of various percentages of anthracite culm 4 and 5 buckwheat coal on sinter production using 65 mesh magnetite.

COAL

Coal is used in MacIntyre sintering as the most economical local source of carbon.

The amount and size of coal used is an important factor in a sintering operation.

As can be seen in Fig 5, 4.0 pct coal produced the best results in sintering 65 mesh magnetite. Both greater and lesser amounts of coal tended to slow the burning time and reduce the production of sinter. Insufficient coal did not produce enough heat for proper sintering and excess coal caused too high a temperature, resulting in slagging and overoxidation with resultant poor sinter. This overoxidation was caused by an oxidizing atmosphere, and while reduction may have taken place during the initial phases of the burning, the final result was excess oxidation.

On the theory that coal used in sintering should be approximately the same particle size as the magnetite, the coal for the sintering tests was ground to 65 mesh. This fine coal, at first, seemed to help sinter production, but sawdust was being used in conjunction with it. As testing progressed, satisfactory results were not obtained, and it was decided to test various sizes of coal. These tests (Fig 6) proved that fine coal, rather than help, did much to deter good sinter production. The No. 4 and 5 buckwheat anthracite culm, as received for 20 mesh sintering, gave excellent results and made it possible to eliminate sawdust from the mix.

The fine coal tended to plug most voids, and made proper air flow impossible. The coarser coal left large voids as it burned, causing lower vacuum and faster burning time.

MOISTURE

The amount of water that can be added to a 65 mesh magnetite "mix" is limited. The "mix" will sinter, if all other proportions are correct, at a moisture between 6 and 8 pct, with the best results obtained at 7.5 pct moisture (Fig 7).

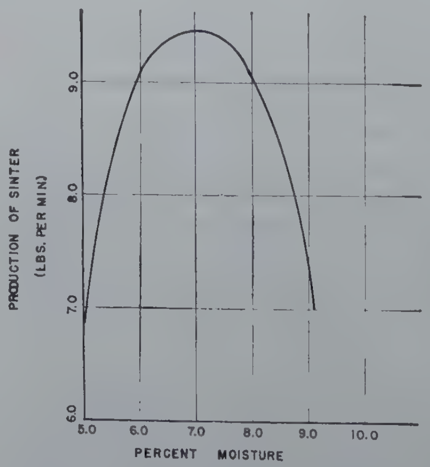


FIG 7—Effect of various percentages of moisture on sinter production with 65 mesh magnetite.

If the moisture is too high, the mix becomes semifluid and it is impossible to draw air through the bed. Thus, ignition is impossible. If the moisture is low, holes tend to form as the material is pulled through the grates. The air, following the path of least resistance, is drawn through the holes and thereby short-circuits the bed. Poor burning results and less sinter is produced.

With 20 mesh magnetite, a wider moisture range is possible. Good sinter was produced with a moisture as low as 3 pct, and as high as 8 pct. However, with 20 mesh magnetite, the percentage of moisture required is closely related to the amount of dust present in the returns. If there were more than 35 pct of minus 35 mesh material in the return fines, it was necessary to use a moisture of 7.5 pct (production with the same mix at 7.5 pct moisture was 10.8 lb per min and at 6.0 pct moisture, production was 9.6 lb per min). If the returns contain less than 35 pct minus 35 mesh material, best results are obtained with a 6 pct moisture.

SLAKED LIME AND MISCELLANEOUS REAGENTS

In an effort to reduce burning time, and thus increase production, slaked lime was added to the charge. A 10 pct increase in production was obtained with the addition of 0.7 pct lime to the 65 mesh mix (Fig 8).

If an excess of lime is added, the charge will burn hot and fast, with little sinter being produced. It is thought that the lime causes an increase in the surface tension of the water. With this higher surface tension, the water would fill less voids in the

Table 2 . . . Effect of Lime on Varying Amounts of Coal

	Production (Lb per Min)	Cycle Time (Min)	Temperature Exhaust Gas Degree C	Quality	Vacuum In. of Water		Per Cent Lime
					Start	End	
With 3 pct coal...	5.5	9.0	520	81.5	61	48	0.75
With 4 pct coal...	11.0	10.0	325	82.2	57	53	0.75

Table 3 . . . Effects of Various Reagents on 65 Mesh Magnetite Sinter

Reagent	Per Cent Reagent	Production, Lb per Min	Per Cent Plus 1 In. Sinter	Quality	Cycle Time, (Min)
Standard		9.0	65.1	78.9	13.1
Lime	0.7	9.9	63.6	78.0	11.5
Detergent	0.005	8.1	64.2	86.0	15.0
Oleic acid	0.05	(would not burn)			
Calcium chloride	2.0	9.7	61.4	74.8	12.0
Pine oil	0.05	5.1	52.2	88.2	20.5
Zinc stearate	0.5	(would not burn)			
Aluminum dust	0.5	5.8	54.8	78.9	18.0

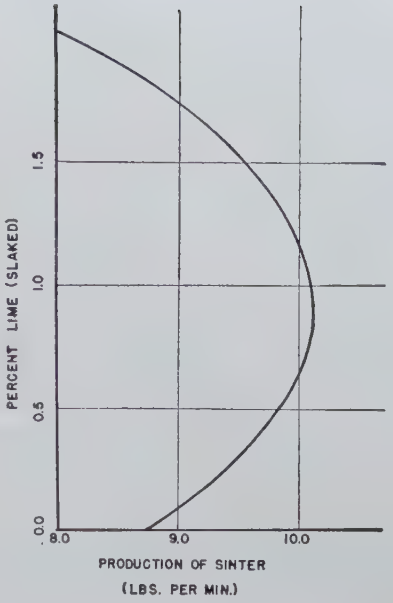


FIG 8—Effect of various percentages of slaked lime on sinter production with 65 mesh magnetite.

mix, thus leaving greater and more numerous voids than possible with unadjusted water. With more numerous voids, a greater volume of air was drawn through the bed, lowering the vacuum, decreasing the burning time, and causing a higher burning temperature. The addition of excess lime increased the surface tension to such an extent that an excess volume of air was drawn through the charge, causing such rapid and hot burning that little sinter was produced.

If the percentage of lime was held constant and the coal percentage dropped, less sinter was formed because of higher burning temperatures. Higher burning temperatures slag the sinter.

The results of Table 2 show that with 3 pct coal and 0.75 pct slaked lime, the mix burned hotter, and gave poorer results than those obtained using 4 pct coal and 0.75 pct slaked lime. This can be explained by the knowledge that a higher percentage of carbon causes excess slagging. This slagging reduces the amount of voids, and this tends to slow the burning rate. This shows, again, how critical the addition of lime would be in actual plant practice. If lime were added in excess, production would drop. If the amount of lime remained constant and the percentage of carbon dropped, production would again drop. Thus, if lime is used in actual plant practice, efficient metallurgical and quantity controls must be provided.

In order to check the surface tension theory, a series of tests were made using the following reagents (Table 3): (1) detergent ("Dreft"), (2) oleic acid, (3) calcium chloride, (4) pine oil, (5) zinc stearate, and (6) aluminum dust.

It is known that a detergent reduces the surface tension of water. Thus, if a detergent is added to the mix, a poorer sinter should result, because with a lower surface tension, the water would fill more voids. The results obtained by test work checked with this idea. When 50 g of detergent were added to the mix, the mix became "putty-like" in appearance and semifluid in nature. The sized return fines were completely disintegrated when they were placed in water containing a detergent. When this mix was added to the pan it was impossible to draw air through it, and thus it could not be ignited.

Calcium chloride, which gives a substantial increase in the surface tension of water, was next added to the mix. The calcium chloride decreased the burning time, but produced a weak sinter. The sinter cake actually expanded during burning when calcium chloride was in the mix. Why this expansion took place is unknown. Calcium chloride could not be used in sinter plant operation because of the large amounts of chlorine gas liberated during the burning; however, it was used in the pilot plant because it was known to increase the surface tension of water.

All other reagents tried gave poor results.

OXIDATION

For a short while during the test work, it was thought that the oxidation of the FeO to Fe_2O_3 was an important factor in strong sinter. To check this point many assays were made on magnetite feed, and sinter produced from this magnetite. As a result of a study of these assays it is thought that oxidation to a certain extent does produce stronger sinter by causing the iron molecules to share oxygen atoms. When oxidation progresses too far, the iron is oxidized completely and no longer shares oxygen atoms, thus producing weaker sinter. When oxidation is low, there is little sharing of oxygen atoms and again the sinter is weak.

Table 4 . . . Effects of Oxidation upon Quality of Sinter

Magnetite			Sinter			Quality
Fe	FeO	Fe_2O_3	Fe	FeO	Fe_2O_3	
57.0	31.04	46.99	56.8	21.78	57.06	75.8
56.7	31.61	45.91	56.0	16.52	61.68	87.8
58.0	32.33	46.97	56.5	9.63	70.05	72.8

Table 4 is representative of the average assays completed, and shows weaker sinter on both the high and low oxidation samples.

LIMESTONE

Because of the cheaper costs of limestone compared to slaked lime, it was thought desirable to check the effects of limestone in the "mix." The limestone was sized from minus 6 plus 10, minus 10 plus 20, minus 20 plus 35, and minus 35 plus 65 mesh. In no case did the limestone help. This was probably caused by the fact that the heat necessary to slake the lime evaporated all the moisture before the limestone was slaked, and thus the effect of the slaked lime on the water was lost.

SAWDUST

Sawdust was helpful when 65 mesh coal was used in the mix. The sawdust

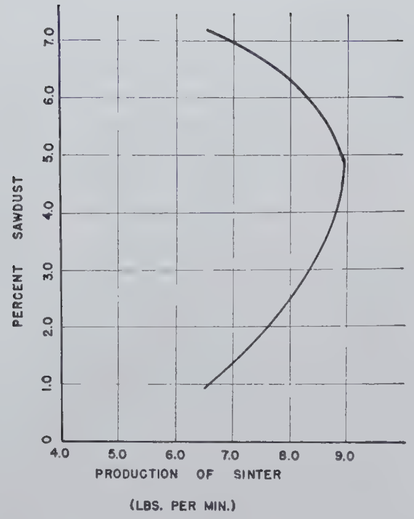


FIG 9—Effect of various percentages of sawdust on sinter production with 65 mesh magnetite.

had a tendency to "fluff" the mix in the pan, and it also made voids for air passage as the mix was burned. Fig 9 shows a high point for sawdust addition. As the percentage of sawdust was increased above 5, the mix burned very hot and fast and sinter production dropped. Sawdust elimination simplified the general problem because of the difficulties that its handling would have entailed if it were needed for 65 mesh magnetite sintering. The use of No. 4 and 5 buckwheat coal eliminated the need for sawdust.

PELLETS

A rotary mixer was used for pilot plant mixing. During the mixing of some batches, pellets were formed by magnetite building up around particles of return fines. The coarser the return fines, the larger the pellets would become. These pellets interferred with sinter production by causing short-circuiting of air around them because they had no porosity. Therefore they were unable to be sintered. The pellets were baked by the heat around them, but crumbled when subjected to the slightest pressure. No figures were obtained and the only method of making these determinations was by visible observation.

In sinter plant operation, pug mills are used for mixing and this problem should not occur. However, if pellets do form, some method to eliminate them must be provided. This does not infer sintering of a completely pelletized charge was undertaken. In all cases the pellets were mixed with unpelletized material, and under such conditions, were detrimental.

DUST

Dust is defined in this test work as material in the return fines, less than 65 mesh for 20 mesh magnetite sinter and minus 200 mesh for 65 mesh magnetite sinter. After it was known that excess fine material would reduce sinter production with 65 mesh material, tests were made to determine what effects "dust" would have on minus 20 mesh material. It was found that if the moisture was increased as the percentage of "dust" increased, the amount of "dust" in the "mix" had little effect on sintering of 20 mesh magnetite.

MIXING

Early in the project, several mixing tests were run using pug mill discharge. When this material was further mixed in the rotary mixer, a sharp production increase was noted. This mixing was checked again at the end of the project, and no increase in production could be attained by further mixing. In some cases production fell off after additional mixing, probably caused by water filling the voids in the return fines. It is thought that the pug mill blades, and other plant conditions were responsible for the poor results obtained during the first testing, but with the pug mills in good shape, as far as blade wear is concerned, a complete mix should be attainable. It was found that a 5 min rotary mix, after the magnetite had been brought to the correct moisture, gave the best results for rotary mixing in the test plant.

PRODUCTION RUNS

When the test work was completed it was necessary to test the best results. For this purpose, several pans were burned in rapid succession to see how the "mix" would react in a hot pan over a period of time. The recommendations made were on the basis of the production run results.

Conclusions

The best quality and quantity of sinter on this test pan were obtained with the following mix:

4.0 pct coal
25.0 pct return fines (minus 10 mesh plus 0)
(at least 18.75 pct minus 10 mesh plus 35 mesh)
70.3 pct magnetite (minus 65 mesh size)
0.7 pct slaked lime.

The average production on the test pan with the above "mix" was 9.9 lb per min or 246 lb per hr per sq ft of grate area. This is slightly higher than average production in the sintering plant pans on minus 20 mesh magnetite.

The pilot plant work indicates that minus 65 magnetite can be sintered in the sinter plant if close control can be maintained over the following items:

1. Adequate amount of bedding material (minus 1 in. plus ½ in. return fines for hearth layer.
2. Sized return fines (minus 10 mesh plus 0).
3. Anthracite coal (size and moisture percentage).
4. Moisture percentage in the "mix."
5. Magnetite (moisture percentage).
6. Percentage of magnetite, coal, lime, and return fines in the prepared feed to the plant.

The results obtained in the sinter testing show that a more accurate metallurgical control on overall operation will be necessary for sintering minus 65 mesh magnetite than is needed in present plant operation for sintering minus 20 mesh magnetite.

Before the present sintering plant can handle minus 65 mesh magnetite, the plant flowsheet will have to be changed to include the conveying, cooling, screening, storing, and weighing of both return fines and bedding material.

Because of the difference between the size of the test pan (2.4 sq ft grate area) further development work will be needed in the sintering plant. A production run of at least two months would be required on minus 65 mesh magnetite to study the effect of the finer mesh size magnetite on the operation and maintenance of the plant.

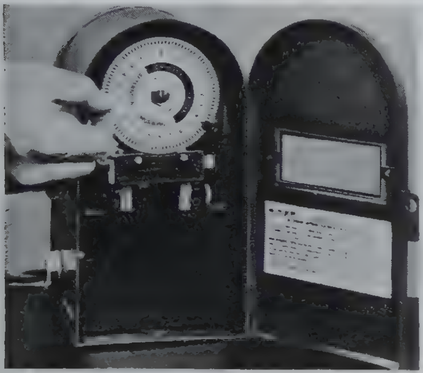
Acknowledgment

The authors wish to thank the following individuals for their suggestions and help in making the sintering tests and for their help in making it possible for them to write this paper: Messrs. Gloyd M. Wiles, Chairman of the Mining Committee, National Lead Co., Frank R. Milliken, Plant Manager, MacIntyre Development, National Lead Co., A. R. Reiser, former Assistant Plant Manager, M. F. Morgan, Arthur G. McKee & Co., John E. Greenawalt, and the following metallurgical personnel of MacIntyre Development; Messrs. Ernest M. Spokes and Elton Geist.

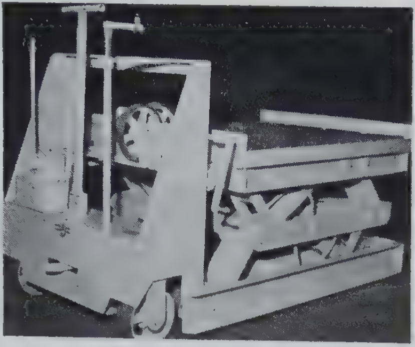
AMONG THE MANUFACTURERS:

Laboratory Clock Switch

A new laboratory day-night clock switch provides a control device through which the technician can preset electrical equipment to be turned on or off as long as a week after the switch has been set. Thus a laboratory furnace, oven or other equipment, requiring long warm-up time, can be turned off automatically at any given time the next morning. The switch operates on 110 volts, 60 cycle a.c. and will control equipment up to 1500 watts current.



A calendar wheel adjustment permits setting the mechanism to repeat the same on-off cycle each day—or to skip any day of the week. It is made by the Fisher Scientific Co., 717 Forbes St., Pittsburgh 19.



Die Handling Truck

Special die handling trucks cut down the time required for changing heavy dies and by eliminating danger hazards, thus justifying their cost, states the manufacturer, Lyon - Raymond Corp., 2421 Madison St., Greene, N. Y. This 16,000 lb. capacity hydraulic elevating die handling truck has been designed for large dies. It is furnished with a platform, 48 in. wide by 57 in. long, the roller top consisting of five rows of rollers placed on 4 in. centers. With the roller top in place, the platform can be lowered to 24 in. or elevated to 38 in. The top can be removed, when the dies are supported by a flat steel top. The hand winch, used to draw dies on the table, is geared to a 96 to 1 ratio and is supplied with 25 ft. of steel cable.

Temperature Indicators

Temperature-indicating products of the crayon and pellet types can be obtained for the first time in the temperature range of 1600 F to 2000 F. Beginning with 113 F, Tempilstiks and Tempil Pellets are now available in 12½ degree steps to 400 F and in 50-degree steps from 400 to 2000 F. A paint form of temperature indicator is available in similar intervals from 113 F to 1600 F. The attached photo indicates a 200 F stick drawn across a warm surface. It has left a glistening liquid streak where the surface is at or above 200 F and a dry chalky mark where the temperature was below 200 F. Another photo, not published here, indicates two pellets, one marked 1950 and the other 2000. The heated surface is apparently between these figures, Fahrenheit since the 1950 pellet has started to melt, the other remaining intact. They are made by Tempil Corp., 132 W. 22nd St., New York 11.

Manufacturers' Publications

A pamphlet on Colmonoy Spraweld wire has been issued by the Wall Colmonoy Corp., 19345 John R. St., Detroit 3. The hard-facing wire is used in the process, "spraywelding," which combines metallizing with welding. The Colmonoy 6 has a hardness of 56 to 61 Rockwell C and is extremely resistant to corrosion, oxidation and abrasion. It can be used in standard metallizing guns.

Equipment for the measurement and control of pH in industrial processing operations is described in a 16-page bulletin, No. 430, published by the Foxboro Co., Foxboro, Mass. Case histories are cited from numerous industrial fields to illustrate the importance of dependable pH control.

The Bristol Co., Waterbury 91, Conn., has listed in bulletin W1805 its various products including recording instruments for temperature such as thermometers, pyrometers and potentiometers; also electric and pneumatic controllers of many kinds.

New data on corrosion resisting high silicon irons, Duriron and Durichlor, are contained in a new publication, Bulletin 113, of the Duriron Co., Inc., Dayton 1, Ohio. Four pages are devoted to chemical processes where the material is suitable.

Men of Industry

Dr. Max Hansen, distinguished German metallurgist, has joined the staff of Armour Research Foundation, Illinois Institute of Technology, as senior metallurgist in the nonferrous section, his initial work to be on titanium, aluminum-beryllium alloys and copper alloy wire. He came to the United States from Germany in 1947 where he was director of research for Durener Metallwerke, in Duren, an organization that introduced aluminum alloys. He is an author of note, an outstanding book being "Constitution of Binary Alloys."

Edward H. Snyder, president, Combined Metals Reduction Co., Salt Lake City, has been elected president of the American Zinc Institute, succeeding Howard I. Young, who has held the office since 1935. Three new vice presidents were elected: Clarence Glass, Anaconda Sales Co.; George Mixter, U. S. Smelting, Refining & Mining Co., and Raymond F. Orr, Athletic Mining & Smelting Co.

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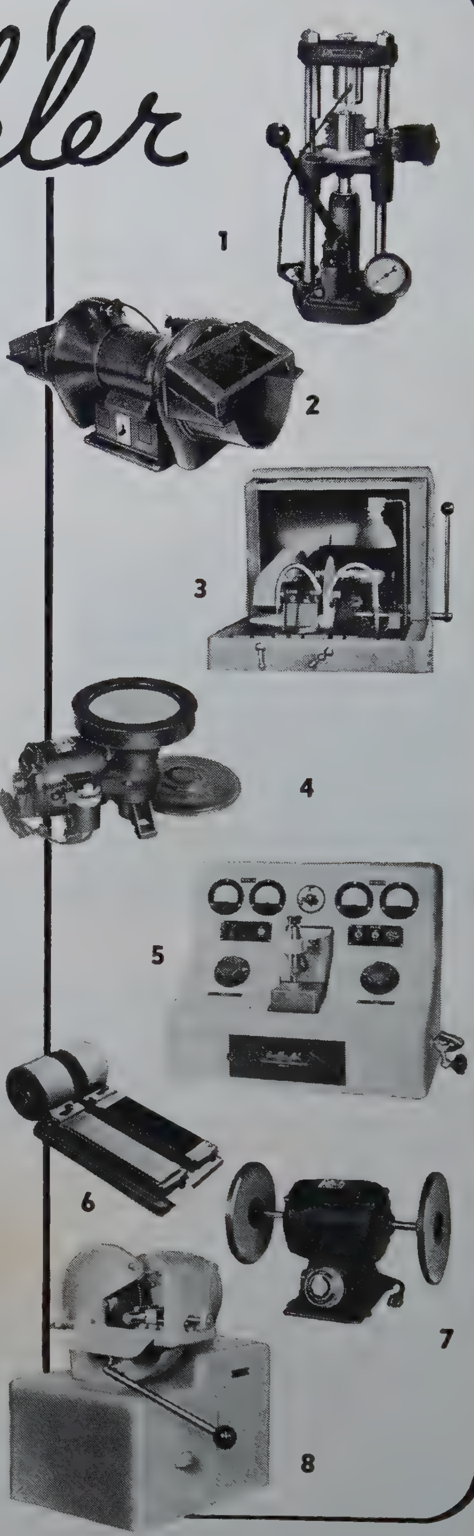

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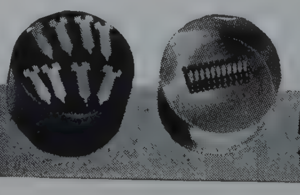

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METALLURGIST, 29. Degree in Met. Engr. Experienced in physical testing, assay work, electrolytic silver-gold production and short period in experimental powder metals laboratory. Desires position in or leading to production work. Midwest or eastern U. S. M-437.

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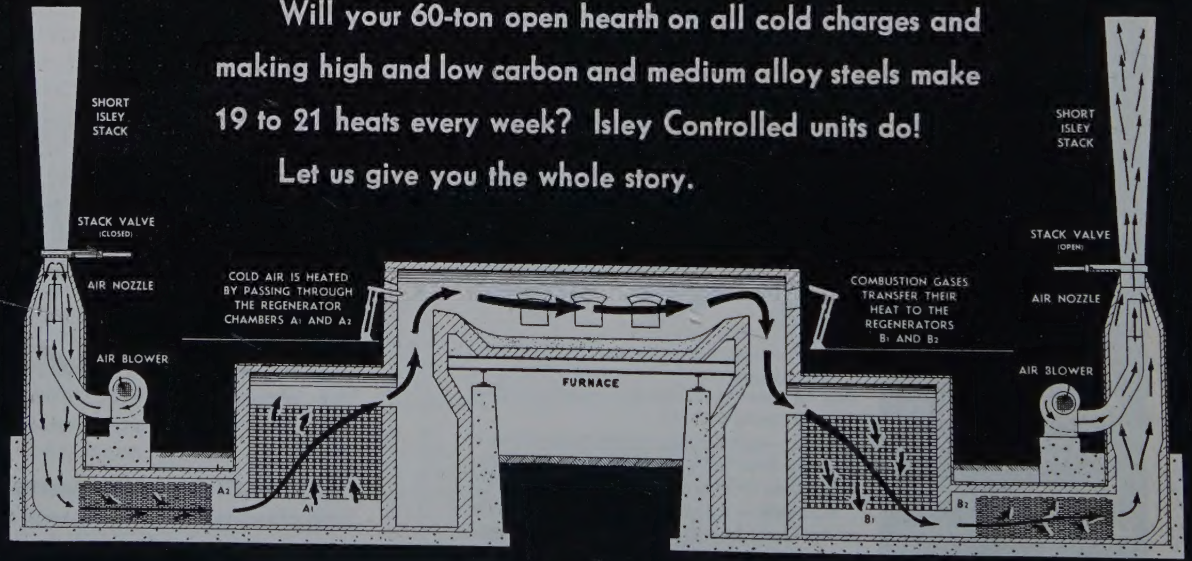
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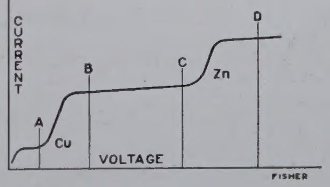

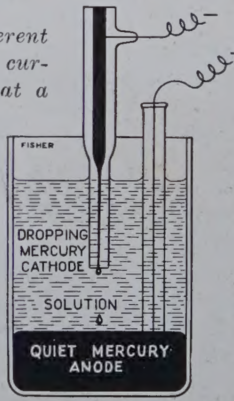

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